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METALS & ALLOYS

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The Magazine of Metallurgical Engineering

INCLUDING

CURRENT METALLURGICAL ABSTRACTS

WORLD'S LARGEST PLATE MILL AT LUKENS STEEL COMPANY, COATESVILLE, PA.

VOLUME 4 AUGUST 1933 NUMBER 8

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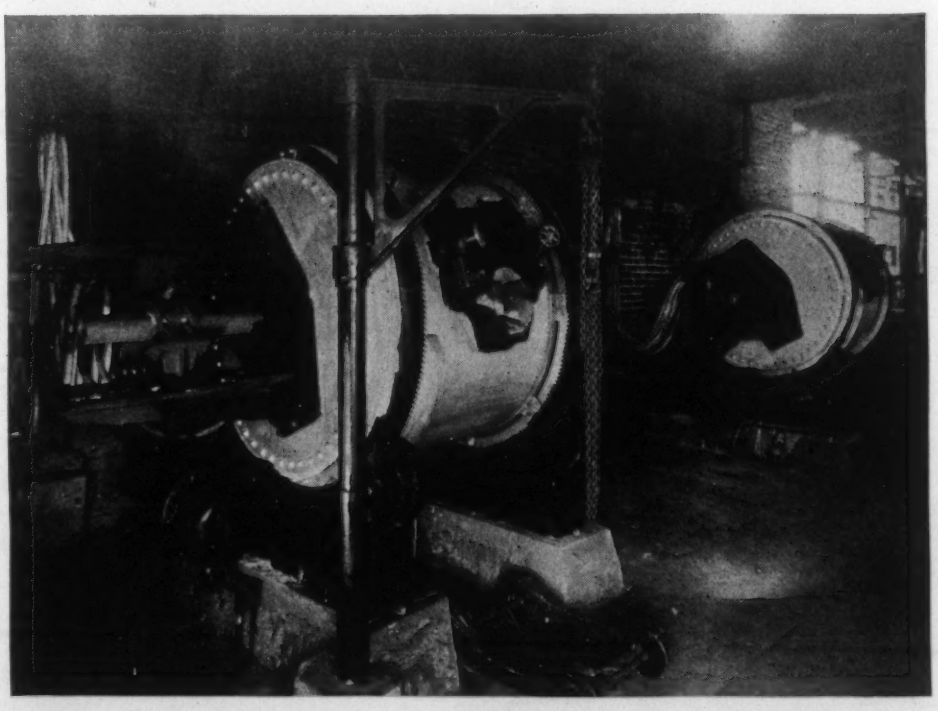

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HIGHLIGHTS

by H. W. GILLETT

High Test Cast Iron

Special cast iron comes in for attention this month. Meyersberg (page MA 244 R5) summarizes the high strength iron from the German point of view. MacPherran (page MA 244 R3) does the same from that of the U. S.

●

Quick, the smelling salts!

Give an idle boiler a sniff of ammonia, if you want to keep it from rusting while unemployed, says Seyb (page MA 245 L7).

●

Wire for Coal Screening Sieves

Körner finds (page MA 246 R1) that wire for sieves in screening coal and coke and in ore classification wear best if made from austenitic manganese steel. Sounds like a rather sweeping conclusion, for the pressure in some types of ore dressing shouldn't be high enough for this to be the case.

●

Silicon and Age Hardening

Silicon is edging into a lot of non-ferrous alloys, usually conferring precipitation-hardening effects. Wilson and co-workers (page MA 247 L2) discuss Cu-Ni-Si and Cu-Co-Si while Ballay (page MA 242 L6) studied the former as did Guillet. Dahl (page MA 256 R10) deals with the straight Ni-Si system. Guillet and Ballay also comment on Cu-Ni-Sn and Cu-Ni-Sn-Si.

●

Electrodeposit Bitumen for Protection

Electrophoresis is used, according to the *Commonwealth Engineer* (page MA 246 R7) to coat the inside of cast iron pipe. You fill it with a bituminous emulsion, drag an electrode through the pipe and the current coats the pipe, just like depositing rubber by electrophoresis.

●

Duplex Rails

Duplex rails with high carbon head and low carbon base, made from a layer ingot are on the market in Germany (page MA 253 L2). Cost is doubled, but laboratory wear tests indicate 5 times the life. Wonder whether service wear will corroborate laboratory tests this time.

DO YOU want to know what metallurgical engineers are saying, the world over? Look in the Current Metallurgical Abstracts. Here are some of the points covered by authors whose articles are abstracted in this issue.

Solder

Cadmium solders (page MA 257 L2) an 80 Pb, 10 Sn, 10 Cd solder is mentioned as "a standard German solder" and said to be as good as 60 Pb, 40 Sn, while Montelucci (page MA 257 L5) speaks a good word for Cd-Zn eutectic solder.

●

Good-bye Cupola?

Editorial comment in the (British) Foundry Trade Journal (page MA 263 R2) says foundrymen will soon consider the cupola obsolete for high strength and alloy irons, replacing cupolas by oil or powdered coal furnaces. Becker (page MA 263 R3) and "Anon" (page MA 263 R6, also page MA 263 R7) speak most highly of rotary powdered coal melting furnaces. See also page MA 265 L9 & 10 and Giolitti (page MA 261 L9).

●

Thermal Expansion of Cores

Not often studied, but Varlet (page MA 263 R5) says it may be a cause of castings not being true to pattern.

●

Stresses in Heat Treated Forgings

Internal stresses in large heat-treated forgings are discussed by Kirchberg (page MA 250 R9). Hollow boring is advocated.

A Rare Topic

But here's a topic on which articles are rare—quenching oils by Krekeler (page MA 255 L5).

●

U. S. vs Germany in Steel Manufacture

Russian observer (Chekmarev, page MA 262 R2) says U. S. is ahead of Germany in methods of pouring steel ingots, but Germany is ahead in utilization of heat, especially in soaking pit practice.

●

High Strength Structural Steel

High silicon, manganese and copper and a little chromium and molybdenum if desired, give a structural steel (St 52) i. e. about 75,000 lbs./in.^{2} tensile, that is finding commercial favor in Germany says Koppenberg (page MA 242 R10).

●

Heat Treat Cast Iron

No month would be complete without some articles on heat-treatment of cast iron so both Heller and Calbiani (page MA 255 L8) summarize its status.

●

Mill Scale Not so Bad

Wrought iron and copper steel are said by Britton & Evans (page MA 271 R1, page MA 271 R3) to stand up better in atmospheric corrosion, not only bare but also when painted, than do ordinary steels, because of more adherent rust coatings. Mill scale is not so bad, say these authors, as long as it isn't broken, if it is broken, its presence is dangerous. While the dark of the moon has apparently not yet been studied in regard to painting of metals, it is found that increased amount of drier helps when paint is applied in wet weather, but harms in dry.

●

Endurance

Gerbes (page MA 251 L5) says endurance of all hardened and un-drawn steels is practically alike because of internal stress and not affected by composition. That's a pretty sweeping conclusion and not likely to be entirely true, though, broadly speaking, he's pretty near right for many hardened steels.

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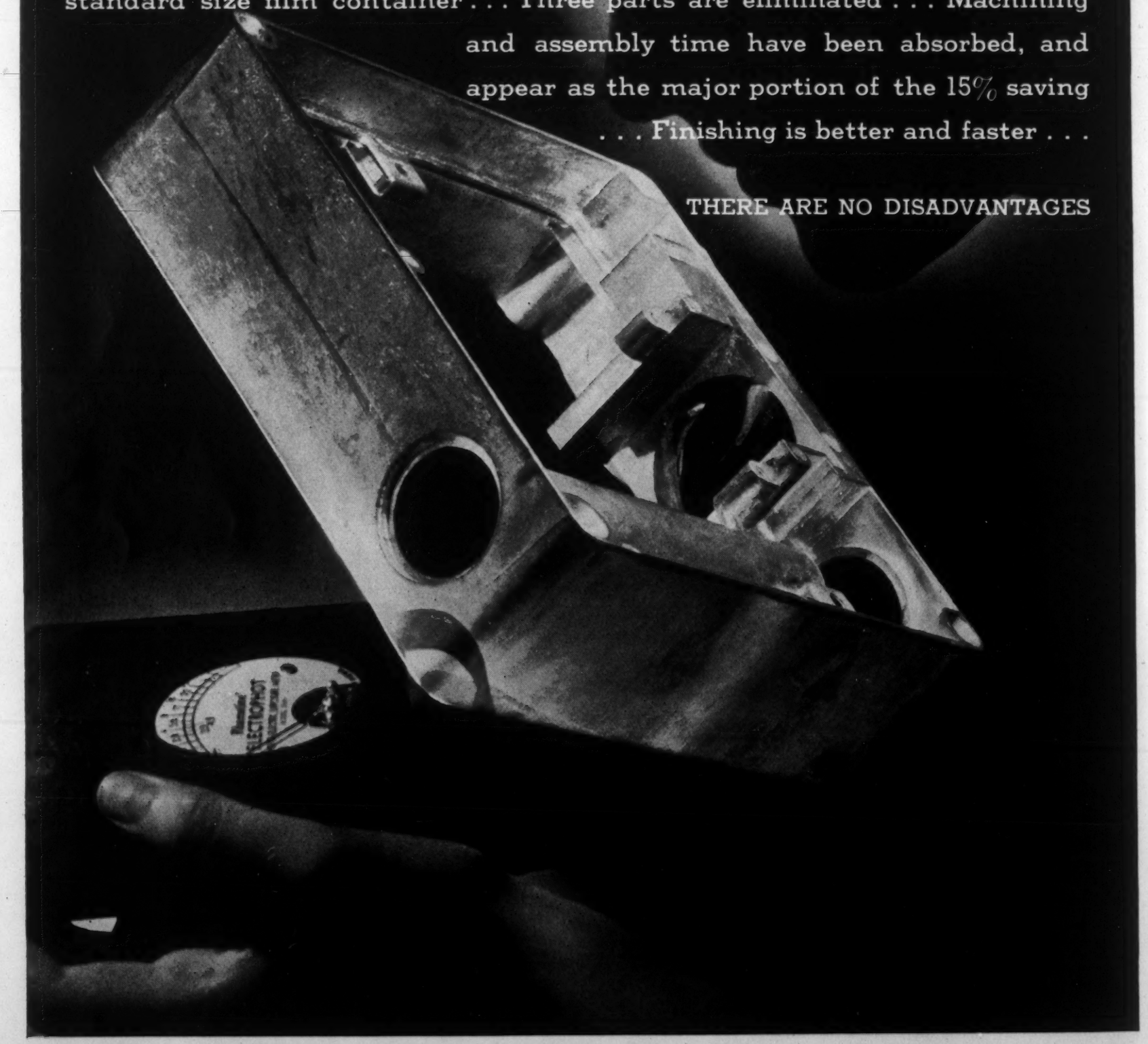

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Editorial Comment

BOUQUETS

THE SESSIONS in Chicago, June 19-30, of the A.F.A., A.S.T.M., A.S.M.E., A.A.A.S., A.I.M.M.E., etc., gave opportunity for many interesting joint meetings. The Cast Iron people made good use of this opportunity. We have already commented on the excellence of the Symposium sponsored jointly by the A.F.A. and the A.S.T.M. Among other more detailed contributions to the joint sessions on Cast Iron that were outstanding were a clear and concise summary of the problem of wear testing of cast iron by A. L. Boegehold, and a comprehensive study of its impact properties by a subcommittee headed by J. T. MacKenzie. The latter has not yet been printed. When it does become available it will be found a mine of information on many things beside impact properties, and worth careful study by makers and users of all grades of cast iron.

In a joint session with the A.S.M.E., the A.F.A. cast iron group continued its campaign of education of the engineer on the range of properties available in modern cast iron.

The cast iron people are working together to find out the facts about their products and put those facts into the hands of the people who need to know them, in extremely effective fashion.

The depression has not brought about any moratorium on technical committee work. There were held during A.S.T.M. week meetings of some 50 metallurgical committees and sub-committees.

In all the sessions of the two weeks of all the societies papers were given or round table discussions held on about 100 topics of metallurgical interest. To those with numerous committee affiliations and varied metallurgical interests as well as some curiosity about the metallurgical exhibits at the Century of Progress, the sum total was pretty much of a 3-ring circus, and a lot that one would have liked to attend had to be left for future study of printed papers and discussions. On the whole, however, the lumping of so many different topics and so many attractions justified the time and place of the meetings.

There was a distinct improvement in the mechanics of handling the technical sessions of both the A.F.A. and the A.S.T.M. Care had been taken to schedule only such a number of papers as could be presented and discussed in the time available if the authors gave them concisely and the session chairmen kept the meeting moving along. In general the authors and chairmen did meet these requirements nicely. As a result the technical sessions were astonishingly well attended in spite of the heat, the poor ventilation of many of the hotel meeting rooms, and the simultaneous attractions at the Century of Progress. Firms who may have hesitated to send representatives to the conventions, for fear they would show up mostly at the Fair and only secondarily at the technical meetings, would have been pleased to note that the metallurgical engineers were distinctly putting business ahead of pleasure.

The A.S.T.M. deserves a special nosegay of its own for simplifying the routine of voting on specifications, saving time while still avoiding anything savoring of steam-roller tactics. The scheme worked so well that it is to be hoped that still further progress can be made in avoiding wearisome formalities.

Finally, the A.S.T.M. is to be congratulated on getting Dr. Gough, the eminent English authority on fatigue, to be present. His Marburg lecture was, of course, the high spot of the meeting from the metallurgical point of view, and will receive careful study as soon as it is available in printed form. His sound and illuminating discussion of the various papers on fatigue of metals was also thoroughly appreciated. Dr. Gough made many friends on his visit just as his colleague Dr. Desch has done on his visits to the United States.

Our largest bouquet is held in reserve for some future date when, if a June convention must be held in Chicago, the session would be held at some hotel that will air-condition the meeting rooms and not merely the dining rooms.—H. W. Gillett

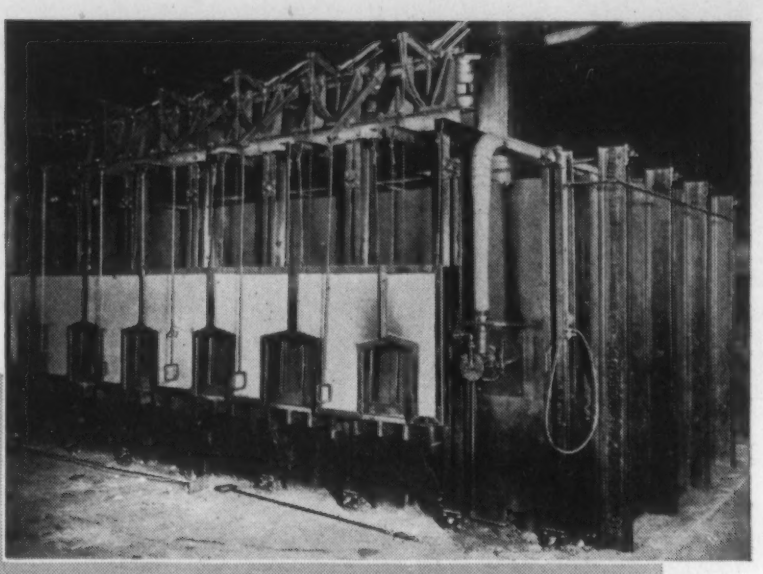
Metallurgical Theses

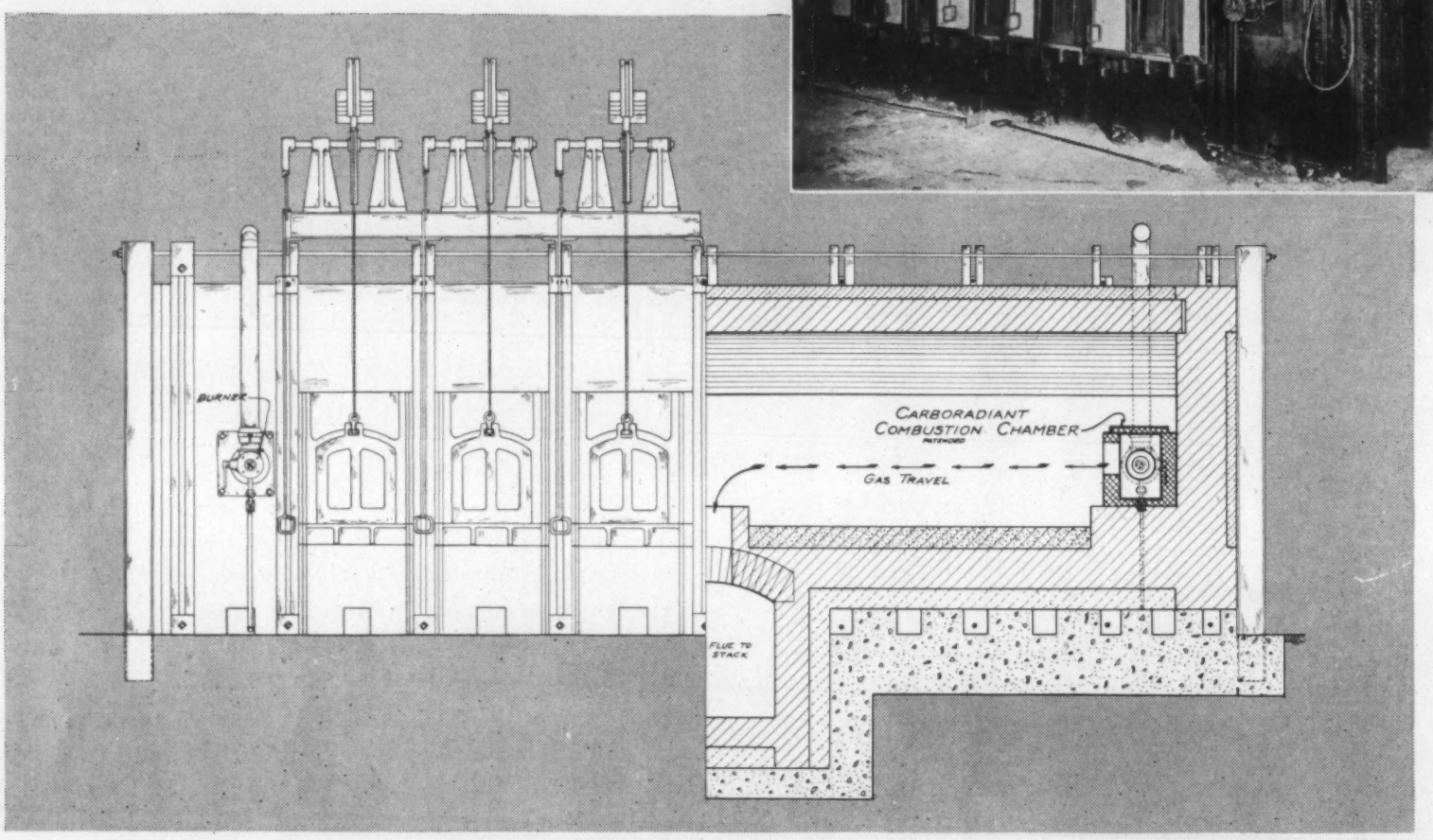
WHILE most metallurgical theses for the doctorate are ultimately published in full or by means of articles giving the gist of their findings, few theses for the master's degree are published. While full publication would seldom be justified, especially when the subject is to be pursued further, yet there are generally items of value in them. Even an occasional senior thesis has some points worth recording.

These theses, while not published, are nevertheless on file in the college libraries and available for public use. They may usually be borrowed on interlibrary loans. They are a part of the metallurgical information that is theoretically available. Practically, their availability is confined to that particular college because others do not know of their existence.

It would appear that these theses deserve abstracting, or at least mention by title, so that those specially interested in their subjects may know of their existence and be able to look them up if they desire. If the professor in charge or the librarian will send copies of such theses to Metals & Alloys on loan, they will be abstracted. Theses of former years that are still unpublished may be included as well as those of the present vintage.—Richard Rimbach

THE PROOF of Performance





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A Few Problems in Non-ferrous Castings*

by Dr. Georg Masing**

THIS is a continuation of the article published in the July number. Dr. Masing here discusses gases in Y-alloys, and deoxidation and structure of copper castings.

3. GASES IN ALUMINUM ALLOYS

We have seen that gas evolution has a marked effect on shrinkage and on segregation. Let us now consider the effect of gases in more detail taking up first the behavior of the "Y" alloy (4% Cu, 2% Ni, 1½% Mg, balance commercial Al) developed by the National Physical Laboratory of England.

This is difficult to cast sound, and a series of investigations have been made in England in the attempt to overcome this trouble, in which treatment with nitrogen, solidification in the crucible and remelting and, later, treatment with chlorine gas, or by CCl_4, $SiCl_4$, $TiCl_4$, etc., carried by a stream of nitrogen has been advocated.

These processes are inconvenient and difficult to carry out in the foundry. Very pure and dry nitrogen is required to avoid the decomposition of such liquids as $SiCl_4$ and $TiCl_4$ and the oxidation of the melt. Hence the process is difficult to use in the rough business of a foundry.

Treatment with metal salts which are solid at room temperature, seems to be much easier, and practical results have confirmed this view. In the foundry of the Siemens-Schuckert works in Berlin the Y-alloy is treated with zinc chloride, although this treatment has become less important recently for reasons which will be discussed later.

In the experimental work leading up to this different salts were introduced by means of a small reversed crucible provided with holes so as to enable the molten salt or the gases to pass slowly into the molten alloy. The alloy was then cast into rods for tensile tests 2 cm. in diameter, 20 cm. long either in metal molds or in dry sand.

To ascertain the degree of unsoundness of the sand castings, their densities were measured and compared with those of the chill cast rods. The difference between the density values of the two was taken as a measure of the unsoundness of the sand casting.

Table 8 shows the influence of different chlorides on the density of the Y-alloy. In the first column, there are given the differences in density mentioned above as a measure of unsoundness, in the next ones the results of tensile strength tests. It will be seen that the unsoundness of 0.103-0.134 is reduced by $ZnCl_2$ to 0.042 as mean value of the same order. But the treatment with $MgCl_2$, $CaCl_2$ and NH_4Cl proved to be useless.

These results show the effect of salt treatment on the soundness of Y-alloy. The salt employed must be reduced by the molten aluminum, giving aluminum chloride, while the metal of the chloride is being alloyed with aluminum. It is obvious that the condition of the formation of aluminum chloride is fulfilled also in the case of chlorine gas or the other liquid chlorides mentioned above. If this is necessary and considered sufficient, it would be expected that treatment with aluminum chloride itself be still better, and though the use of $AlCl_3$ offers difficulties, on account of its high volatility, it has proved to be as successful as other chlorides, as it reduces the unsoundness to the value of .045, as may be seen in the table.

It is interesting to note that the unsoundness is of but little account in the tensile strength properties of the alloy. This seems to be a general behavior of the aluminum alloys. Unsoundness in them is not so objectionable from the standpoint of strength, elastic limit, elongation and fatigue limit as for many other materials, or as we would expect it to be.

Of all the chlorides experimentally tested, $ZnCl_2$ has been found most suitable. Most of the other chlorides evaporate more easily than does $ZnCl_2$ and are more liable to dissociation by the humidity of the air into hydrochloric acid and hydroxide.

If $ZnCl_2$ is introduced in the shape of larger pieces on the surface of the molten aluminum alloy, a rapid reaction begins, gas bubbles separate from the molten metal and explode in contact with the air. On the surface of the molten aluminum, a dry skin is formed which is quite different from the film covering the surface without addition of $ZnCl_2$. This skin consists of $AlCl_3$ and ZnO and can easily be skimmed away. Although $ZnCl_2$ itself fumes

*Condensed from a lecture delivered at the Carnegie Institute of Technology.

**Metallurgist, Research Laboratory, Siemens-Werke, Berlin-Siemensstadt, Germany.

Table 8. Influence of Treatments with Different Chlorides on the Density of Y-Alloy Sand Castings.

Salt	Unsoundness (difference in density)	Elastic limit 0.003% lbs./in.²	Yield Point 0.2% lbs./in.²	Tensile Strength lbs./in.²	% Elongation in 8″
No Treatment	0.134 0.103				
$CaCl_2$	0.137			24,200	1.6
$MgCl_2$	0.206 0.058	11,500	23,000	26,500 29,200	1.0 1.2
NH_4Cl	0.153			26,200	1.2
$ZnCl_2$	0.060 0.050 0.050 0.022 0.041 0.032	11,900	22,800	30,200	1.3
	0.042				
$CdCl_2$	0.041	10,800	21,300	29,700	1.0
$PbCl_2$	0.041				
$SnCl_4 + N_2$	0.030	11,600	25,600	29,600	1.0
$SiCl_4 + N_2$	0.025	13,000	25,400	30,500	0.8
$AlCl_3$	0.025	11,900	22,900	31,300	0.9

considerably at 700°-800°C. no appreciable fuming occurs during the reaction with aluminum. The reaction lasts some minutes. When it is over, the alloy is skimmed in the usual way and can then be cast.

In the first experiments, we used 2% of $ZnCl_2$. Later it was found that 0.5% or even 0.25% is sufficient. When working with large melts (50 lbs. or more), it is better to treat the alloy 2 or 3 times with smaller quantities of salt, than once with the whole amount of the salt.

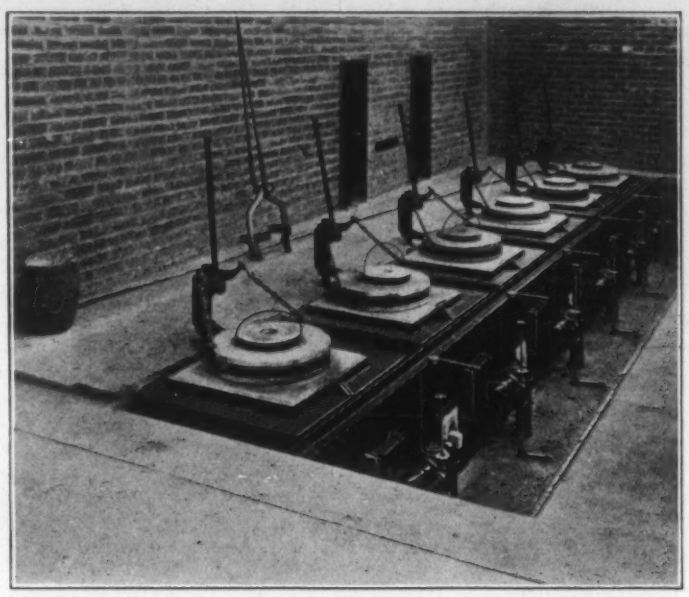
Zinc chloride is to a certain extent reduced by molten aluminum. Thus far it has not been possible to detect any objectionable influence of this small content of zinc in the alloy. Treated with $ZnCl_2$, this alloy, as sandcast, has always been found to be superior to the chill-cast Y-alloys not treated with $ZnCl_2$. This holds more particularly for the mechanical properties.

What is the cause of the unsoundness of alloys such as the "Y" alloy?

When treated with $ZnCl_2$ or with nitrogen a combustible gas is separated from the molten metal. This gas can only be hydrogen. This is shown by the fact that hydrogen led through a molten alloy gives rise to serious unsoundness. If the action of $ZnCl_2$ consists in the removal of hydrogen, how can the pressure of this hydrogen be explained? It must be assumed that the alloy contains not only dissolved hydrogen, but also to a certain extent, gaseous hydrogen in the shape of small bubbles. These bubbles are probably prevented from rising to the surface by the oxide layers floating within the metal. By an addition of $ZnCl_2$ and by stirring, these layers are coagulated and can no longer prevent hydrogen bubbles from rising to the surface of the metal; they are evolved and burn in the air. The coagulating agent for Al_2O_3 is probably $AlCl_3$.

There is another proof that the molten "Y" alloy generally contains hydrogen or, more generally, gas-bubbles in its interior, for it is possible to avoid unsoundness of the castings by maintaining the molten alloy for some time (say 1 hour) . . . at a higher temperature (900°-1000°C.). Under these conditions the gas bubbles are capable of reaching the surface.

It is a very interesting fact that pure aluminum as well as 8% Cu-alloy and the 13% silicon alloy cannot be improved as much by the treatment with $ZnCl_2$, as the "Y" alloy. Several alloys of aluminum are extremely unsound when cast. Tullis has found that this is caused by the presence of certain elements, such as magnesium



Crucible Furnaces at the International Silver Company. (Courtesy W. S. Rockwell Company.)

or calcium. Such additions have been designated by him as "disturber" elements. He assumes that in pure aluminum or in its alloys not containing "disturbers," the whole amount of the gas (predominantly hydrogen) is actually in solution, but that this equilibrium is "disturbed" as soon as one of the "disturbers" is added. Only such disturbed alloys are adapted for the treatment with chlorine compounds. There can be hardly any doubt that the "disturbance" of the equilibrium condition consists in the separation of small hydrogen gas bubbles from the molten aluminum. This effect is probably caused by the decreased hydrogen solubility or to the capacity of the aluminum to hold the hydrogen in a supersaturated solution.

The action of chlorides and other compounds of chlorine can therefore be readily understood. Only the gas bubbles suspended in an oxide nest in the interior of the molten aluminum are removed by this treatment, and only such bubbles lead to the grave unsoundness observed in alloys such as "Y" alloy. The unsoundness of materials such as commercially pure aluminum or alloys rich in aluminum seems to be of another origin, viz., the separation of the dissolved gas during solidification. For the removal of hydrogen in this state, the chlorine treatment is not suitable and other means must be chosen.

The chlorine treatment (as we shall call the treatment

Fig. 9

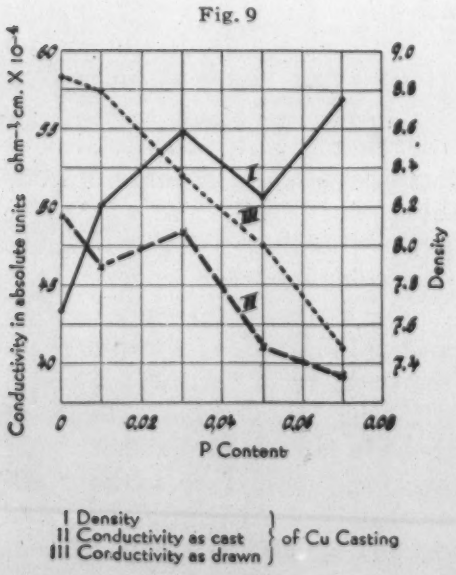


Fig. 10

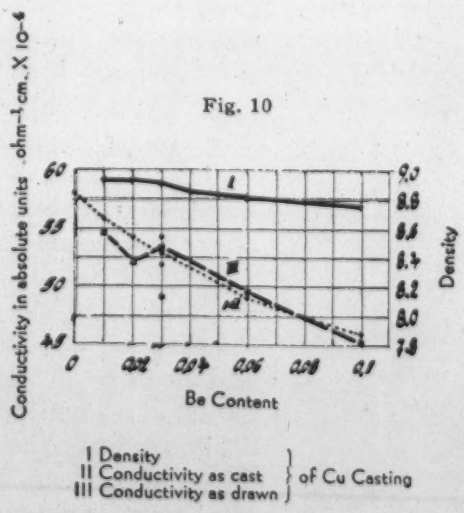
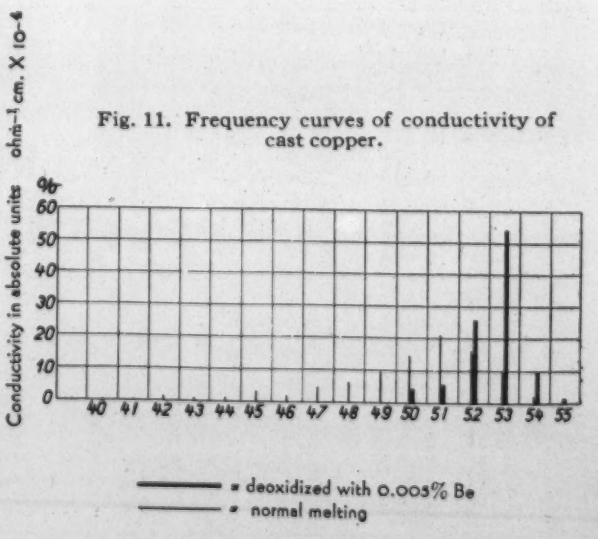


Fig. 11. Frequency curves of conductivity of cast copper.



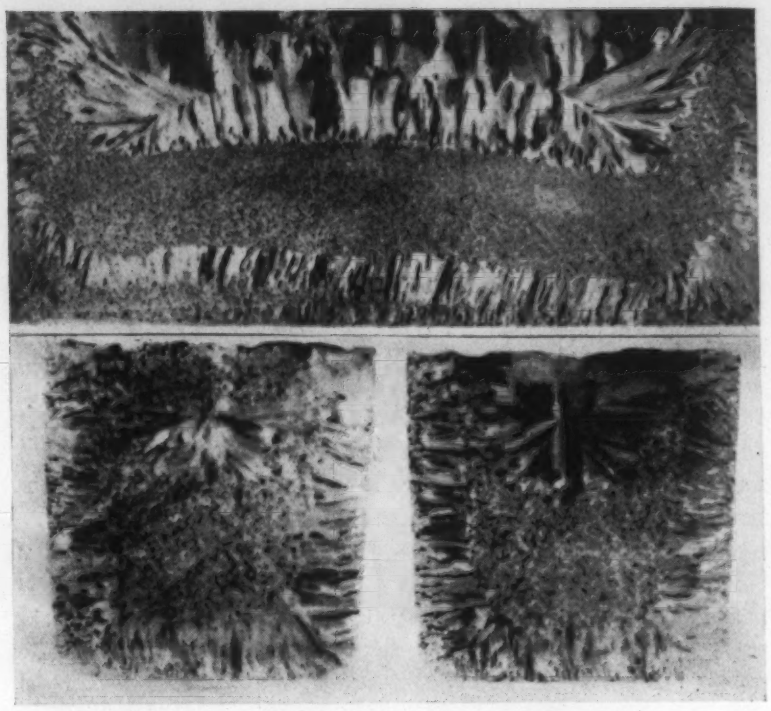


Fig. 12. Crystal structure of a copper wire bar.

with all the chlorine compounds and salts) in most plants is usually not applied to the Y-alloy, and I have mentioned above that this treatment has gradually become less important. This is because of a change in quality of commercial aluminum. When repeating the laboratory experiments with the Y-alloy in 1930-1932, it was found that, even without any treatment, unsoundness was rarely obtained. Unsoundness was produced only when virgin aluminum was deliberately treated with hydrogen in the molten state before being chill cast and employed for the preparation of the "Y" alloy. Apparently, therefore, commercial aluminum contains less hydrogen nowadays than formerly.

The author is of the opinion that the great advances made on the previous practice of the production of the Y-alloy and of many other alloys of this class are, in one way or another, due to the improvement of the aluminum quality at least to the same extent as to other factors, as for instance, the improvement of the casting conditions themselves.

The comments on the $ZnCl_2$ treatment may not be new to the majority of American readers, for I am informed that such treatment has been practised in American foundries for thirty years or more. The research data herewith presented, however, are largely new and are presented for this reason.

4. THE CASTING OF COPPER

The commercial production of sound castings of pure copper offers great difficulties. It had been thought for some time that the reason for unsoundness, especially of copper sand castings, lay in the presence of sulphur together with oxygen leading to the evolution of sulphur dioxide at the solidification point. However, we now know that the greatest danger is the simultaneous presence of hydrogen and oxygen (in the form of cuprous oxide) in the molten metal. The water vapor formed by reaction between these elements is evolved, together with the excess of hydrogen, during solidification, thus producing porosity in the casting.

The means generally applied for avoiding porosity is the deoxidation of copper, in most cases with phosphorus. The oxygen then combines with the deoxidizer and cannot react with hydrogen. It seems that a certain amount of hydrogen is not objectionable in the absence of oxygen for it can be maintained in a supersaturated solid solution in the cast copper, thus preventing the production of unsoundness.

But phosphorus lowers the conductivity of copper considerably, to a value as low as only 75% of the theoretical conductivity of pure copper. For many electro-technical purposes this conductivity is much too low. The decrease in the electrical conductivity of copper by phosphorus is far greater than would be expected theoretically from the amount of phosphorus in the copper. Indeed one finds in technical literature that a content of 0.13% P lowers the electrical conductivity to only 72.7% of the pure copper value. By interpolation we should find for the content of 0.02% P technically used the value 95% instead of the 75% mentioned above.

The strong influence of phosphorus upon the conductivity must therefore be conditioned by causes other than its constitution alone. This conclusion is supported by laboratory experiments, in which the electric conductivity and the density of small sand cast copper rods of 28 mm. in diameter having various contents of phosphorus were measured. After being measured the rods were cold-rolled and drawn to wire of 2 mm. in diameter and annealed at 600°C. for one hour. Afterwards the conductivity was measured again. The results are shown in Fig. 9.

The conductivity in the cast condition is lower than in the rolled and drawn wire. Furthermore, there is a close relation between this difference in the conductivities and the density of the material: the lower the density, the greater the difference between the conductivities in the cast and cold-worked conditions. That sand cast copper deoxidized with phosphorus is far from having the normal density has been very often observed. It seems to be characteristic of phosphorus that it is not capable of removing minute unsoundness in copper though it avoids the formation of the larger holes which make the casting useless practically. It is very probable, that this low density accounts for the low values of electrical conductivity found in sand cast copper deoxidized with phosphorus.

There are many other deoxidizers which help to produce sand castings of copper densities which approach the true density. Excellent results can be obtained with lithium, calcium, magnesium, aluminum, and beryllium, having density values as high as 8.9. In view of the very low content of these metals retained in the alloyed copper an electrical conductivity of 90-95% in the sand cast condition can easily be obtained, although all these metals lower the conductivity. But another difficulty appears here: all the metals, with the exception of beryllium, give rise to the formation of "oxide-nests" on the surface of the casting thus rendering it unserviceable.

In our experiments we have not been able to overcome this difficulty and have therefore developed the deoxidation of copper with beryllium.

It is a very striking feature, that the surface of the castings treated with beryllium is as completely satisfactory as with phosphorus, and that in spite of the close resemblance of the oxide of beryllium with those of

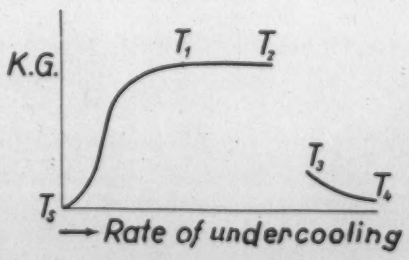


Fig. 13. Velocity of crystallization.

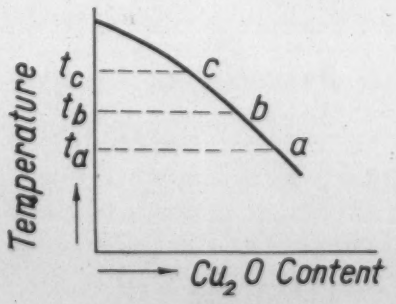


Fig. 14. Germination and undercooling in wire bars.

Teeming a Sterling Silver Ingot at the Handy & Harman Plant.

magnesium and aluminum, the latter give poor surfaces. This difference in the behavior of oxides so similar as these has not as yet been satisfactorily explained, although it is an easy enough task to propose hypotheses.

Fig. 10 gives the results of measurements of the conductivity and density of copper deoxidized with beryllium. The density is high. The conductivities of the castings and of the cold worked and annealed specimens differ but little.

There are two further means for avoiding unsoundness in copper castings: first by superheating to about 1400°C. and second by the addition of boron suboxide. This last deoxidizer has been developed in the United States; so far as I know it is not being applied in Europe. In laboratory experiments it is possible to obtain sound castings with good conductivity by means of boron suboxide, when superheating copper say to about 1300°C. Table 9 shows some results obtained by an addition of boron suboxide, and also by heating the metal to 1400°C. before casting without addition.

Table 9. Deoxidation of Copper by Superheating and by Addition of Boron Suboxide.

Deoxidizer	Impurity (addition)	Density	Conductivity (Absolute unit 58 = 100% approx.)	Heating Temperature
Boron suboxide 1%	Cu_2O 1%	8.94	50.1	low superheating
Boron suboxide 1%	Cu_2O 1%	8.94	55.2	1200°C.
Boron suboxide 1%	Cu_2S 0.5%	8.92	54	1300°C.
Without deoxidizer	Cu_2O 1%	7.80	44.4	not superheated
Without deoxidizer	Cu_2O 1%	8.95	55.3	1400°C.

The temperature required for obtaining a reaction of boron suboxide with the impurities in the copper specimen obviously accounts for the contradictory statements found in technical literature concerning its usefulness. When copper is sufficiently superheated, boron suboxide is an excellent deoxidizer, one which does not alloy with copper and therefore does not decrease the conductivity. But its application requires temperature measurements on the molten copper, generally not an easy task; the question of its practicality must, furthermore, depend on its price.

Superheating to about 1400°C. is also a reliable means to secure sound copper castings. But it obviously decreases crucible life, and it requires careful temperature measurement. For these reasons and perhaps for others, it is not as reliable a means for treating molten copper as the addition of beryllium, which does not require superheating of the melt.

The beryllium deoxidizer generally employed for commercial deoxidation is an alloy of 90% Cu and 10% Be. This alloy is brittle enough to be broken into small pieces if necessary. The amount of beryllium required for complete deoxidation depends largely upon the melting conditions. It is obviously necessary to melt the copper most carefully so as to avoid the taking up of oxygen as far as possible. In two plants with which I am acquainted, the first employs an addition of 0.012% Be to the copper. The melting and working conditions are the same as when phosphorus is used as deoxidizer. The conductivity of copper sand castings averages about 91% of that for pure copper, but it sometimes reaches 95%. The lowest value found as yet is 87%. In the other plant the melts were prepared in 220 lb. crucibles; in this case, it has been possible to obtain good results with only 0.005% Be. Fig. 11 shows the statistical distribution of the conductivity figures for 250 melts deoxidized by superheating (light lines) and of 97 melts with addition of 0.005% Be (heavy lines). Experience proves that the superheating method is not quite reliable. If we require a conductivity higher than 50 units = 87% we see that with beryllium 100% of the castings fulfill this condition, while without beryllium, about 28% of the castings have lower values. If the limit of conductivity will be fixed at 48 units = 83%, there will still be a loss of about 15%. This lack in complete reliability necessitates the control of every melt by measuring the conductivity of a sample rod. This is quite unnecessary in the case of beryllium-deoxidized copper. The costs of both processes will not be compared because the author's figures are only valid for German conditions, but it can easily be calculated that the deoxidation with beryllium is somewhat cheaper than that by superheating. Working with beryllium is much easier since it requires neither temperature nor conductivity control and since the melting process proceeds more rapidly because of the lower temperature.

It may be mentioned that the application of sinkheads for high conductivity sand castings in copper has proved to be impossible even in the presence of beryllium. When calculating the costs of deoxidation, it must be borne in mind that the output is only 35-40% and that the costs rise correspondingly.

Most of the problems so far discussed are present in the manufacture of copper wire bar.

The crystal structure of a copper wire bar is shown in Fig. 12.

There are four distinct crystallization zones. At the surface of the metal mold lies a zone of fine crystal grains. These crystal grains show very little preferred orientation. With increasing distance from the surface of the casting there appear columnar crystals which increase in size. In this part of the casting the orientation is fairly uniform, the crystals show the so called transcrystallization. Then, quite abruptly, there again appear equiaxed crystals orientated at random; these equiaxed crystals become larger and larger until the point is reached where the last of the melt solidifies.

The first two stages of crystallization can be easily understood and involve no problems. The liquid metal poured into a metallic mold is at first quenched at its surface. There occurs crystal germination on a large scale. The crystals, germinated in a melt at a fast rate, do not show preferred orientation. These crystals grow in the direction away from the surface at different velocities, as determined by their orientation. Those crystals having the greatest crystallization velocity perpendicular to the mold will grow the more rapidly and suppress the other crystals, giving rise to the zone of columnar crystals. In this zone we, therefore, have no appreciable crystal germination, but a considerable linear growth.

But why is this columnar stage of crystallization interrupted by another, in which there is evidently a new germination? Genders explains it as follows: During the crystallization of the columnar part of the ingot the cooling rate is so high that the melt crystallizes immediately at the contact with the growing columnar crystals; new germination occurs when the crystallized part of the ingot has become thick enough, so that the heat flow is lowered, and the melt, poured turbulently into the mold is, here and there, capable of breaking off the very weak ends of the growing crystallites. These breaks are carried away with the melt and act as crystal nuclei during the subsequent crystallization process.

This is a very interesting theory, but it is based on a somewhat questionable assumption. The crystal particles broken off by the melt can act as nuclei only if they are not again remelted by the molten metal. It is possible for them to remain solid only if the temperature of the metal at the edge of the crystals lies below the melting point of the metal. But its temperature is, of course, higher than that of the crystallizing front proceeding into the melt.

We must therefore conclude that the temperature of the ends of the growing crystal needles or skeletons is below the melting point. This conclusion is in contradiction with the theory of linear crystal growth suggested by Tammann, and needs some inspection.

If one introduces a supercooled pure organic liquid into a glass tube and puts a crystal nucleus therein, the crystallization proceeds at a velocity depending on the increasing rate of supercooling. Tammann obtains the well-known curves shown in Fig. 13.

While such curves have been discussed as giving the true dependence of the linear crystal growth on temperature, Tammann has shown that this point of view is not correct. The heat of crystallization is evolved during the solidification process and the temperature of the crystal border where this heat is evolved is far from that of the previous supercooled temperature. Tammann concludes from the shape of curve $T_s T_1 T_2$, especially from the horizontal part $T_1 T_2$, that the temperature of the crystal border must be constant from T_1 to T_2 and furthermore that it must be a characteristic temperature, indeed the temperature of the melting point. The heat necessary to raise the melt up to the melting temperature, owing to the higher supercooling rate, is, of course, greater at T_2 than at T_1. This heat is controlled by the number of crystals growing together at the border. While at T_1 there are still comparatively few of such needles, since the rest of the melt crystallizes later between the needles, the whole melt crystallizes practically at once at T_2. At the lower temperatures of curve $T_3 T_4$, the heat of crystallization is no longer sufficient to reach the melting point, the temperature of the crystal border remains below this temperature, and the linear crystal growth falls to far lower values.

By calculating the heat development it can be shown that the constant temperature of the crystal border between T_1 and T_2 must not of necessity be the temperature of the point of fusion, but that it may be lower. It now seems certain that the temperature at the front of a solidifying crystal needle of many organic substances is far below the temperature of fusion. It is, in fact, difficult to understand how it should be otherwise. The rate of linear growth is a reaction velocity and must be zero at the point of equilibrium, i.e., at the point of fusion. From the principal point of view a finite crystal growth can, therefore, only occur at temperatures below the temperature of fusion, although the amount of its deviation from this temperature may be quite different for different bodies. From this point of view the conclusion which we have drawn is quite sound.

General View of the Handy & Harman Melting Shop.

But there is still another point of view with respect to copper ingots which may explain the occurrence of the zone of germination inside the columnar zone. Technical copper generally contains cuprous oxide, and cuprous oxide during the solidification is being concentrated in the molten liquid at the center of the ingot, while the primary crystals are practically pure copper. At the border of the primary crystals the melt must, therefore, gradually become richer in cuprous oxide. Since crystallization takes place at a very fast rate, the homogenization of the liquid by diffusion or convection currents must be incomplete, and just at the front of the crystal skeleton of pure copper the liquid will be far richer in cuprous oxide than in the center of the melt. The temperature at these spots will correspond to, say, point a, Fig. 14. In the neighborhood of the melt it will be higher and correspond to, say, t_b. But it is possible that the concentration of the liquid will here be in accordance with point c. The melt will be supercooled here, and the fragments of the copper crystals carried there by convection will not be dissolved, but will at once show further growth.

This theory also explains the abrupt beginning of the zone of second germination behind the columnar zone. The amount of cuprous oxide in the melt adjacent to the copper crystals will increase continuously with proceeding crystallization. The condition of germination will only be fulfilled if the influence of the concentration gradient exceeds that of the temperature gradient.

Hence we see that all the peculiarities inherent in the crystal structure of a copper wire bar may be understood. But if we try to discriminate between different possible causes and to estimate their influence, we fail. This failure has a general reason which is characteristic of much research in physical metallurgy. In discussing the different problems of casting we have seen, in connection with almost any question, that we must confine ourselves to a purely qualitative treatment. Thus, so far as our theories remain qualitative, it is very difficult to choose between different possible explanations. For this reason many theories of physical metallurgy are as yet quite uncertain and we may not be surprised that physicists in general do not like to deal with metallographic questions; the development of quantitative theories with the help of exact measurements is of fundamental importance for further advances in physical metallurgy.

Some Physical Properties of **Commercial Thorium**†

by J. G. Thompson*,**

DETERMINATION of some of the physical properties of thorium, prepared by electrolytic and calcium-reduction processes, was undertaken. Serious difficulties were encountered in a number of attempts to determine the melting point. The results of the melting point determinations were not satisfactory but indicate that the generally accepted value for the melting point of thorium may be high. Crystal structure, density, hardness, electrical resistivity, thermal expansion and thermal e.m.f. against platinum were determined for one or both kinds of thorium.

INTRODUCTION

Thorium was discovered by Berzelius early in the 19th century, but to-day it is still considered a rare element about which little is known. The only commercial sources of thorium are monazite sands containing from 2 to 10% thorium oxide, and the supply of these sands is limited. For many years interest in thorium or its compounds was confined to the oxide which was used in incandescent gas mantles. The high melting point and refractory properties of purified thorium oxide led to the use of this material, particularly in the fused form, in the preparation of small refractory shapes for laboratory investigations at high temperatures.[11, 39] The presence of small amounts of thorium in metallic tungsten has been shown to be desirable in the production of lamp filaments as the thorium tungsten alloy is less susceptible to grain growth during heat treatment than is pure tungsten.[30] The presence of thorium increases the electron emission of tungsten, consequently thorium alloys have been used in X-ray and radio tubes.[14, 16, 27] Other uses for thorium or its compounds have been proposed[14, 19, 20, 33] but none of these minor uses appear to have commercial significance at present.

The use of alloys of thorium stimulated interest in the metal itself. Although a number of methods for the production of metallic thorium or its alloys have been proposed,[3, 4, 5, 15, 18, 23, 24, 27, 38] it is believed that recent production of thorium has been accomplished either by the calcium-reduction method[15, 17, 22] or by electrolytic methods such as that of Driggs and Lilliendahl.[36, 40, 41]

Scattered information concerning the properties of metallic thorium is found in the literature,[8, 12, 15, 21, 25, 27, 28, 34] with particular attention devoted to discussion of the possible superconductivity of thorium.[26, 29, 32, 35, 37] Values for the ordinary physical properties, as reported by different observers, are not always in good agreement, in particular there is considerable uncertainty regarding the melting point. The Bureau of Standards therefore undertook to determine the melting point and certain other physical properties of commercial thorium.

MATERIAL FOR THE INVESTIGATION

Calcium-reduced thorium in the form of powdered metal and rods or wires 10, 5, 1 and 0.4 mm. in diameter, was supplied by the Westinghouse Lamp Company of Bloomfield, N. J., and electrolytic thorium in the form of 1 mm. and 0.4 mm. wire, a fused button, and powdered metal was supplied by the Kemet Laboratories Company, Inc., of Cleveland, Ohio. Subsequently, when additional material was needed, 10 mm. rods of both calcium-reduced and electrolytic metal were supplied by the Westinghouse Lamp Company. The samples were used for the determination of different properties but, owing to the limited amount of material available, it was not always possible to use both electrolytic and calcium-reduced metal for determination of the same physical property. All of the various specimens were used as received.

Purity of 99.7 or 99.8%, after heat treatment, has been claimed[15] for thorium produced by the calcium-reduction process; Fe was reported never to exceed 0.1%, Si was not determinable, and the presence of C could not be established by the usual analytical procedures. Purity comparable with that of the calcium-reduced material has been claimed[36] for the product of an electrolytic process, the impurities in the electrolytic metal being reported as C 0.005%, Si 0.01% and Fe 0.005%. Details of the electrolytic process used by the Kemet Laboratories Company are not available, but it was reported by the maker that the powder, from which some of the specimens for this investigation were compacted, contained 99.7% Th with 0.02% Fe and 0.06% Na present as impurities.

Owing to the limited amount of material available, systematic analysis of all the samples was not possible. A single chemical analysis of one of the 10 mm. rods of calcium-reduced metal indicated that 0.055% C, 0.02% Si, 0.058% Fe, 0.078% Ca, and a trace of Al were present. Na, Ti and V were not detected. Spectrochemical analysis indicated the presence, in the electrolytic metal, of Ca, Co, W and Mo and further indicated variations between different specimens of calcium-reduced metal, particularly in the Ca content.

It is not possible, therefore, to state definitely the purity of either the calcium-reduced or the electrolytic metal used in this investigation. Spectrochemical analyses and the fact that wires of calcium-reduced metal were decidedly brittle in comparison with wires of electrolytic metal, suggested a difference in composition between the 2 types of metal. The erratic behavior of a number of specimens in the subsequent determination of properties seemed to confirm this and in addition to indicate non-uniformity of specimens, particularly of calcium-reduced metal. However, it was considered that the specimens represented commercially available material and the determination of properties was undertaken on this basis.

PROPERTIES OF THORIUM

*1. Melting point.**** Widely divergent values for the melting point of thorium are found in the literature. The International Critical Tables[12] record a value of 1845° C., without reference to the work from which this value was derived. Marden and Rentschler[15] reported 1842° C. ± 30° as the average value for the melting point of calcium-reduced thorium and cited values

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**With the coöperation of various members of the Bureau Staff, as indicated in the text.

***By Wm. H. Swanger, H. T. Wensel, F. R. Caldwell and J. S. Acken.

of 1450° C.[1] and 1745° C.[2] reported by previous investigators. The melting point of thorium is listed as above 1700° C. in one of the current handbooks.[13] Values of 1450° and 1500° C. also have been observed* although there was some question on the part of the observer as to the accuracy of the methods employed. The determination of the melting point of thorium is rendered more difficult by the tendency of the metal to react with many refractory materials and with elements such as C, O and N.[15]

In the present investigation some preliminary attempts to melt thorium in an Arsem furnace were not successful. In one experiment, with rapid heating, a melting point of about 1690° C. was indicated but there was no arrest on the subsequent cooling curve. In another run, with slower heating, there was no arrest during either heating or cooling. Additional attempts to melt thorium in vacuo in alumina crucibles, or supported on alumina blocks, likewise were not successful. The failure of these experiments can be ascribed to the tendency of hot thorium to react with C, Al_2O_3 and O, as reported by Marden and Rentschler.

An attempt was made to melt a small ingot immersed in barium chloride to prevent oxidation of the thorium during heating. The tip of a platinum to platinum-rhodium thermocouple, in a porcelain protection tube, was immersed in the barium chloride bath close to the thorium ingot. The temperature of the bath was raised until the platinum wire of the thermocouple melted. The ingot did not melt but was appreciably corroded by this exposure to barium chloride at a temperature in excess of 1750° C.

Attempts were made to melt small pieces of calcium-reduced thorium supported on a platinum ribbon which was heated by the passage of an electric current. The pressure in the apparatus was reduced to less than 0.001 mm. before heating was started, to avoid oxidation of the thorium. When the temperature of the ribbon had been raised to about 1400° C. the thorium and platinum alloyed to form a small molten drop which penetrated the ribbon. Subsequently it was found that the temperature of formation of this molten alloy depended upon the rate of heating. With slow heating the molten alloy formed at temperatures appreciably below 1400° C. These experiments further indicate the reaction tendencies of hot thorium.

Attempts to determine the emissivity of thorium by sighting a pyrometer alternately on the thorium and on the platinum strip yielded such varying results that a definite value for the emissivity could not be assigned.

In the next experiments pieces of

*Private communication from H. S. Cooper of the Kemet Laboratories, Inc.

wire 1 mm. in diameter and approximately 7 cm. in length were suspended between water cooled clamps in an evacuated glass globe. The wires were heated by the passage of an electric current, and temperature observations were made by sighting an optical pyrometer into a hole 0.15 mm. in diameter, drilled radially in the wire to a depth of 0.5 mm. Attempts to determine emissivity by simultaneous observations in the hole and on the surface of the wire again yielded widely variant results. With such shallow holes, even of small diameter, only approximate black body conditions can be obtained and in the absence of emissivity data the observed temperatures cannot be corrected to true temperatures. However, it is believed that the emissivity correction under these conditions probably would not exceed 20° or 30° C.

Fig. 1. Thorium melted in a thorium oxide crucible. Dark areas on the light background are thorium which has diffused into thorium oxide. The light areas on the dark background represent diffusion of oxide into the metal. Unetched. Magnification 100X.

Various experimental difficulties were encountered in several runs, particularly if slow rates of heating were employed. Some of the wires swelled during heating, sometimes to such an extent that the sight hole was closed. Other samples developed hot spots and melted through at points removed from the sight hole. However, in approximately half of the runs, operations proceeded in a normal manner and at least one reading was obtained while the sight hole was filling with molten metal. These "good" runs yielded quite consistent results; 1712°, 1723°, 1733° and 1739° C. for calcium-reduced thorium and 1695°, 1672° and 1685° C. for electrolytic metal. The last value, 1685° C., is an average of 4 readings (from 1680° to 1695° C.) obtained while melting was visible in the sight hole.

Attention next was turned to the possibility of using specimens larger than the 1 mm. wires for the melting point determination. Marden and Rentschler reported that thorium could be melted without contamination in slip cast crucibles of thorium oxide. Consequently, thorium oxide was chosen as the refractory for the present investigation and crucibles were constructed according to the method of Swanger and Caldwell.[39] Samples 15 mm. long were cut from the 10 mm. rods of thorium and a 2.5 mm. sight hole was drilled axially in each specimen for approximately 2/3 its length. Each sample, in a thorium oxide crucible, was heated in vacuo by high frequency induced currents. This method and arrangement of apparatus have been used successfully

in determining melting points above 1500° C., e.g., the melting point of rhodium.[31]

Numerous difficulties were encountered in these melting point determinations by the crucible method. A specimen of metallic thorium once melted in a pressed thoria crucible, could not be remelted below 2000° C. This suggested contamination of the melt by the refractory material and this suspicion was confirmed by microscopic examination after an ingot, previously melted, was reheated to about 2000° C. in an attempt to remelt the metal. It was found that enough metal had penetrated into the crucible wall to impart distinct metallic appearance and properties to the oxide crucible. The results of microscopic examination of a section through the crucible wall and ingot, given in Fig. 1, show that the metal penetrated into the crucible and, in turn, the metal was contaminated by oxide, presumably from the crucible. Apparently metallic thorium dissolves thorium oxide to a considerable extent at temperatures near the melting point of the metal. Cooling curves or redeterminations on a single specimen, therefore, could not be made. A single heating curve for each specimen was the best that could be obtained.

Difficulty was also encountered in the tendency of the melts to pick up C from oil vapor diffused back from the vacuum pump. The pronounced odor of the early melts indicated the pick-up of C and chemical analysis of one of the fused buttons showed 0.11% C present, in place of the original 0.06%. However, the installation of a mercury diffusion pump and a liquid air trap between the melting chamber and the oil-sealed vacuum pump eliminated this contamination by C vapors.

Variations in the pressure within the melting chamber during heating indicated liberation of gas either from the metallic specimen or from the furnace tube and refractories. Consequently the furnace and contents were preheated at approximately 800° C., by means of a resistance furnace, until the furnace pressure dropped to 0.001 mm. or less. The resistance furnace was then replaced by a high frequency coil and heating by induction was begun.

In spite of all precautions it was necessary to discard the results of a number of runs, on account of difficulties resulting from the evolution of gases or metal vapors from the samples at temperatures below the melting point, or from the swelling and puffing of certain samples, which was sometimes so pronounced that the sight hole was closed or distorted so that observations could no longer be made. The evolution of gas or vapor within the ingot at temperatures below the melting point of thorium is illustrated by the section of an ingot of electrolytic thorium shown in Fig. 2. The swelling was sufficient to distort the top of the ingot from the original flat surface to the condition shown. The remains of the sight hole, visible at the center of the top of the ingot, show that the metal had barely reached the melting point. This swelling and puffing, particularly noticeable in the case of the calcium-reduced material, may be associated with the C content of the metal, as Marden and Rentschler[15] reported that the presence of carbide caused difficulty when the metal was heat-treated, "a puffing and splattering takes place, spoiling the contour of the sample and making it quite unsatisfactory for mechanical working." Another possible explanation is that Ca vapor was evolved from and throughout the metal at temperatures near the melting point and that this caused or contributed to the puffing and distortion of the specimens. The presence of vapors in the furnace tube was definitely observed in a number of runs, and in some cases were so dense that pyrometer readings, at temperatures approaching the melting point, could not be obtained at all.

Occasionally a determination proceeded in an orthodox manner so that it was possible to obtain a reading while the sight hole was filling with molten metal. Two such runs yielded values of 1697° and 1687° C. for electrolytic thorium and 4 runs on calcium-reduced metal yielded values of 1734°, 1726°, 1720° and 1700° C.

Fig. 2. Metallic Thorium distorted by gas evolution at temperatures below 1700° C. Magnification 3X.

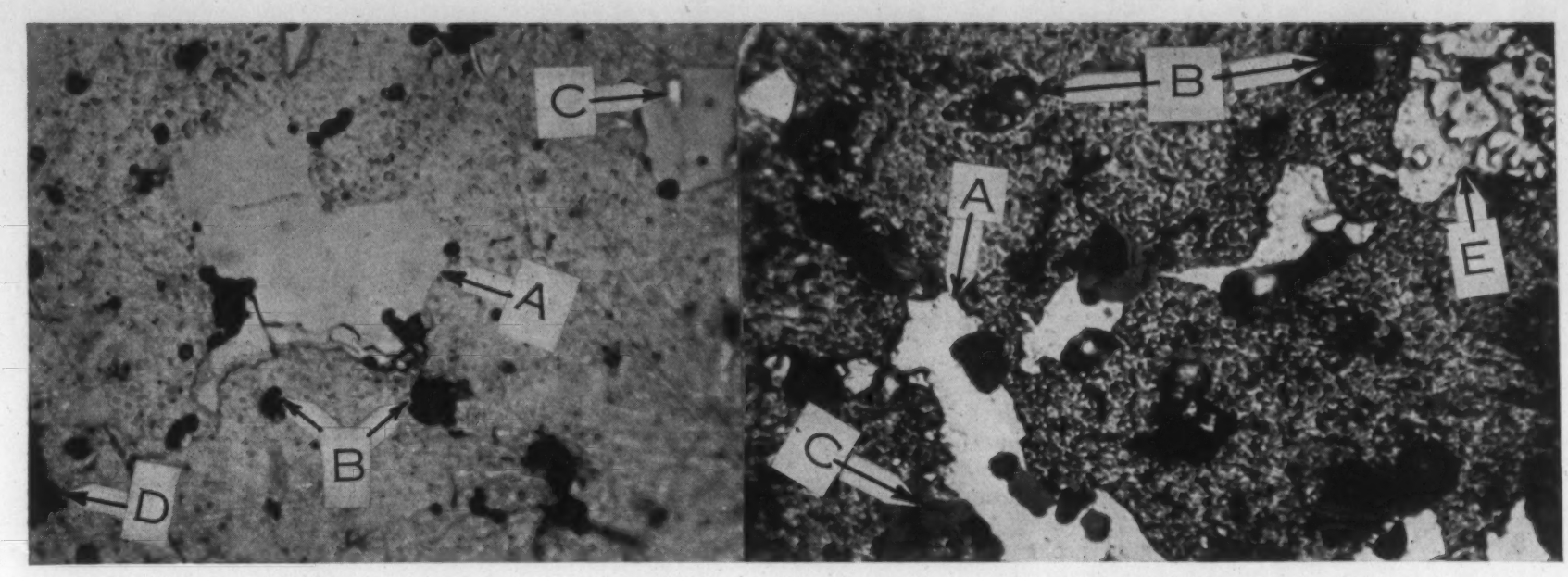
Several attempts were made to avoid the interference by evolved vapors through the use of a covered crucible of thoria, sighting into a small thoria tube which passed vertically through the center of the metal specimen. This type of crucible is not intended for use with small metallic samples where the thermal capacity of the metal is small in proportion to that of the crucible and sight tube and in most of these experiments there was no interruption in the heating curve to indicate a melting point. However, one run yielded a value of 1746° C. for calcium-reduced metal.

These experiments exhausted the original supply of metallic thorium. Subsequently, the Westinghouse Lamp Company furnished additional material, in the form of 10 mm. rods, of both electrolytic and calcium-reduced metal. By the time these additional specimens of metal were received, a supply of beryllium oxide had become available and this refractory was used thereafter instead of thorium oxide. The examination of crucible and contents after a melt of thorium in beryllium oxide indicated that there was little or no reaction between crucible and metal. The metallic ingot could be separated readily from the crucible; a thin, dark-colored skin which usually adhered to the beryllium oxide was the only visible evidence of any action between metal and refractory.

Four determinations on electrolytic thorium, in beryllium oxide crucibles, sighting into a small hole drilled in the metal specimen, yielded values of 1651°, 1670°, 1670° and 1673° C. Swelling and puffing of the ingots occurred but not to an extent great enough to prevent observations. No satisfactory determinations could be obtained on the new specimens of calcium-reduced metal. Spectrochemical analysis indicated the same general order of purity, with somewhat increased Ca content, as compared with the original specimens. The micro-structure of the new material is shown in Fig. 3, the different constituents being identified from the descriptions of Davenport.[21] With this new material, however, vapor evolution was worse than before and fogging of the furnace tube was so severe that no determinations with an open crucible were possible. The use of covered crucibles was attempted but was unsuccessful.

A reliable value for the melting point of either electrolytic, or calcium-reduced thorium cannot be derived from these experiments. The experiments clearly showed that metallic thorium and thorium oxide react readily at temperatures near the melting point of thorium, and further showed that the presence of thorium oxide in metallic thorium raises the melting point. These experiments indicate that metallic thorium cannot be melted in contact with thorium oxide, without contaminating the metal and that subsequent determination of the melting point of this contaminated metal would yield results higher than the true melting point of metallic thorium.

In view of the experimental difficulties the results of any one determination, or of any one method, cannot be accepted as reliable. However, it may be more than a coincidence that the "best" runs, by several different methods, indicate melting points of approximately 1680° and 1730° C. for the electrolytic and calcium-reduced metals respectively. Higher values were obtained only when the melts were contaminated. The single exception was the run in which thorium was heated, to the melting point of platinum, (1773° C.), in a bath of barium chloride and in this run the thorium was severely corroded. These low melting points for thorium, as compared to the accepted value of 1845° C.,[12, 15] might be due to the presence of impurities which lowered the melting point of the Bureau's samples, although these samples were considered to be relatively pure. Another possibility is that the melting point of 1845° C. was obtained on material which contained some thorium oxide. Redetermination of the melting point of metallic thorium appears to be desirable with particular emphasis resting on



Calcium-reduced Thorium

Electrolytic Thorium

Fig. 3. Constituents present in Commercial Metallic Thorium. Unetched. Magnification 1200X. A—Carbide, B—Oxide, C—Orange colored constituent, D—Void, E—Thorium-thorium carbide eutectic.

purity and uniformity of specimen material and on the maintenance of purity during the melting point determination.

*Density.** The density of the specimens of metallic thorium ranged from 11.1 to 11.49 g./cc. depending upon the previous treatment of the specimen. The density of a 10 mm. diameter rod of calcium-reduced metal, sintered and swaged, was 11.32 g./cc. at 25°C. A small button of fused electrolytic thorium had a density of 11.1 g./cc., but the density of a sample of 1 mm. diameter wire of the same material was 11.49 g./cc. at 25°C. These results are in substantial agreement with those of previous investigators.[12, 15, 21]

The computed density, as calculated from the X-ray crystal structure of thorium discussed in the following section, is 11.61 g./cc.

*Crystal structure.*** The crystal structure of thorium was reported by Bohlin[6] to be face-centered cubic with a lattice parameter of 5.12 A.U. Subsequently Hull[7] reported 5.04 A.U. for the lattice parameter.

In the present investigation X-ray determinations were made on both powdered and solid metal. Thorium is highly absorbent to X-rays, as indicated by its high atomic number. Consequently, the powder specimens were diluted with 10 parts of flour, according to the method of Davey.[9] Tubes of lead free soft glass, about 0.5 mm. in diameter, were half filled with the mixtures of metal and flour, the mixture was covered with a small plug of cotton and the remainder of the tube was filled with 200 mesh sodium chloride. Patterns of thorium and sodium chloride were thus obtained side by side on a film, the sodium chloride bands serving as a means of correcting the thorium spacings for errors arising in locating the zero image and also correcting for dimensional changes in the film. In the first experiments the tubes containing the flour and metal powder were carefully evacuated before submitting to the X-rays. It was found that evacuation of the tubes did not affect the X-ray pattern, and consequently was unnecessary. In addition to the powdered metal specimens, some determinations were made on solid metal, by the "reflection" method.

The 3 best films for the calcium-reduced metal yielded results as follows:

Film No.	Lattice Parameter
145a	5.089 A.U. ± 0.006
168	5.091 A.U. ± .003
154	5.092 A.U. ± .002
most probable value =	5.091 A.U. ± .004

Calcium-reduced thorium was used in all 3 experiments, powdered metal in films 145a and 168, solid metal in 154. In 145a, the tube containing the specimen was evacuated, in 168 the tube was at atmospheric pressure. In both cases the metal powder was diluted with 10 parts of flour. In 168 a mixture of 1 part sodium chloride and 1 part flour was used, in 145a sodium chloride alone was used.

Several films obtained from specimens of electrolytic thorium yielded results almost identical with the results for calcium-reduced metal.

Hardness.† Hardness determinations were made on cross-sections of a 10 mm. rod of sintered and swaged calcium-reduced metal. The results for this metal, as received, were as follows:

Scleroscope (average of 5 readings)	28
Baby Brinell—6.4 kg. load; $\frac{1}{16}$ in. ball; 30 sec. (average of 2 impressions)	78
Rockwell "B" (average of 4 impressions)	43

The latter value is considerably lower than the Rockwell "B" values of 70 or more previously reported,[15, 21] for cold-worked thorium.

The same specimen was heated for 15 minutes at 700° to 800°C. in vacuum and a decrease in hardness resulted as follows:

Scleroscope (5 readings)	25
Baby Brinell	74
Rockwell "B" (3 readings)	29

Electrical resistivity.‡ Marden and Rentschler[15] reported that the resistivity of calcium-reduced thorium at 20°C. varied from 13 to 18 x 10^{-6} ohm-cm. depending upon the diameter of the wire and the amount of working. In the present investigation the resistivity of a sample of electrolytic thorium was found to be 18.6 x 10^{-6} ohm-cm. at 20°C. This determination was made on 1 mm. diameter wire, previously referred to as the specimen which exhibited the greatest density.

The average temperature coefficient of electrical resistance, $\frac{Rt - Ro}{tR_o}$, was determined for electrolytic thorium. The fundamental coefficient, for the range 0° to 100°C., was 0.0038. The coefficient for the temperature range —183° to 0°C., was 0.0036.

Attempts to determine the resistivity and the temperature coefficient of resistance of calcium-reduced thorium wires yielded erratic results apparently on account of the physical structure of these wires which were noticeably brittle and non-homogeneous. Marden and Rentschler[14] reported values of 0.0021 between 0° and 1730°C. and 0.0034 between 22° and 135°C., for calcium-reduced metal.

*Linear Thermal Expansion.*** Rentschler and Marden in a preliminary announcement,[10] reported the average coefficient of linear expansion of thorium, between 0° and 100°C., as 12.3 x 10^{-6} and in a later paper[15] as 0.000117. The latter value was perhaps intended for 11.7 x 10^{-6}.

*By E. L. Peffer. **By F. Sillers, Jr.
†By Wm. H. Swanger & J. S. Acken.

‡By F. Wenner, A. R. Lindberg & F. R. Caldwell.
**By Peter Hidnert & W. T. Sweeney.

In the present work the coefficients of linear expansion of calcium-reduced thorium, in the form of a 5 mm. diameter swaged rod, were determined for several temperature ranges, as recorded in Table 1.

Table 1. Thermal Expansion of Calcium-Reduced Thorium

Temperature Range	Average Coefficient of Expansion/°C.
°C.	x 10–6
20 - 60	11.1
60 - 100	11.4
20 - 100	11.3
100 - 200	11.9
20 - 200	11.6
200 - 300	13.1
20 - 300	12.1

*Thermal Electromotive Force against Platinum.*** The thermal e.m.f. of wires of calcium-reduced thorium, against the Bureau's platinum standard No. 27, was determined for temperatures between 0° and 1300°C. The cold junction of the couple was maintained at zero, the hot junction was maintained in a vacuum (at a pressure of 0.001 mm. of mercury). In the case of one specimen the hot junction was carried through a number of successive heating and cooling stages, from room temperature to 1200° to 350° to 1200° to 350° to 1350°C. Determinations of thermal e.m.f. were made at convenient intervals throughout this program, the results being plotted as an e.m.f.-temperature curve. The fact that all of the observations fell within 0.05 millivolts of the curve indicates the thermal stability of the specimen. Values for the thermal e.m.f. at even temperature intervals, taken from the plotted curve, are presented in Table 2.

Table 2. Thermal e. m. f. of Calcium-Reduced Thorium against Platinum

Temperature	E.m.f.	Temperature	E.m.f.
°C.	mv	°C.	mv
0	0.00	700	—0.21
100	— .13	800	+ .22
200	— .26	900	+ .87
300	— .40	1000	+1.73
400	— .50	1100	+2.80
500	— .53	1200	+4.04
600	— .45	1300	+5.42

The sign indicates the direction in which the current will flow at the *cold* junction, i.e., if the e.m.f. is positive the current at the cold junction will flow from the thorium to the platinum.

Summary

The values obtained for certain physical properties of thorium are summarized in Table 3. In this table, the absence of any explanatory symbol indicates that the value applies to both calcium-reduced and electrolytic metal. The symbol (Ca) means that the indicated value refers only to calcium-reduced metal; similarly, (El) refers to electrolytic metal.

The results of the melting point determinations were not satisfactory. The most consistent results were obtained by a wire method, the accuracy and reliability of which are not known. In attempts to employ a crucible method, difficulties resulted from the tendency of hot thorium to react with some of the crucible materials; from the evolution of metal vapors from the hot thorium, particularly from the calcium-reduced metal, at temperatures approaching the melting point; and from the varying behavior of supposedly duplicate specimens. However, the results obtained indicate that the accepted value of 1845°C. for the melting point of thorium may be high and should be confirmed by additional determinations.

The remaining properties in general are in substantial agreement with such previously reported results as are available.

Table 3. Summary of Physical Properties of Thorium

Melting point	Determinations unsatisfactory	
Lattice parameter	5.091 A.U. +.004	
Density, g./cc.	11.1 to 11.49	
Density, calculated from lattice parameter, g./cc.	11.61	
Hardness (Ca)		
Scleroscope	28-25	
Baby Brinell	78-74	
Rockwell B	43-29	
Electrical resistivity at 20°C., ohm-cm. (El)	18.6 x 10–6	
Temperature coefficient of electrical resistance/°C. (El)		
0° to 100°C.	0.0038	
—183° to 0°C.	.0036	
Coefficient of linear thermal expansion (Ca)		
20° to 60°C.	11.1 x 10–6	
20° to 300°C.	12.1 x 10–6	
Thermal e.m.f. vs. Pt, Millivolts, (Ca)	100°C.	—0.13
	300	— .40
	500	— .53
	700	— .21
	900	+ .87
	1100	+2.80
	1300	+5.42

**By H. T. Wensel & Wm. F. Roeser.

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Metal Microscopy at High Temperatures*

The Iron-Carbon Diagram and the Solubility of Oxygen in Solid Iron

By Hans Esser and Heinz Cornelius

THE utility of the equipment and method described in this article are apparent. The conclusions drawn from the application of the method to the iron-oxygen system deserve thoughtful consideration, but further work will be required for a complete solution of the problem.

Since the authors found that oxide inclusions grew larger on heating, the smaller ones "amalgamating" with the larger ones, and since this would probably occur though solution of oxide from small particles and precipitation, from the solution, upon larger particles, their conclusion as to insolubility of oxide does not appear to be necessarily borne out by their own experiments. The hypothesis of bodily migration of insoluble particles needs definite proof of each particular instance before its acceptance.

Hence the suggested modifications of the Fe-O diagram, and the conclusion that oxygen does not enter into precipitation hardening phenomena in iron, should still be considered as tentative, till more evidence is available.

The usual methods for the determination of the changes (allotropic transformations, solubility- and decomposition-phenomena), which take place in metals and alloys on change of temperature are chiefly based on observations of the temperature-coefficient of certain physical properties. The first attempt to observe such phenomena directly in the microscope was made by P. Oberhoffer.[1] In his apparatus the section became covered by a thin film (perhaps oxide), so that observation at high temperatures was not possible.

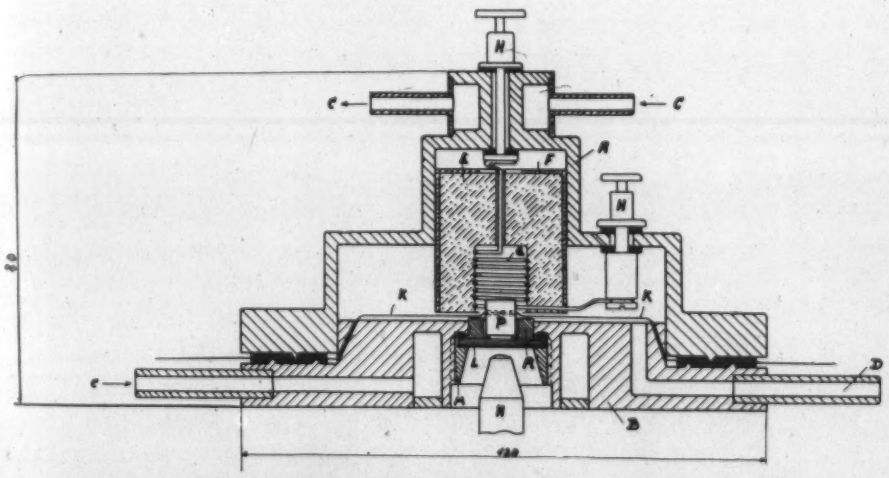

Fig. 1. Apparatus for Metallographic Investigation at High Temperatures.

An apparatus which permits the metallographic examination of sections up to temperatures of about 1100°C. in a comparatively simple way, is described below. Since the heating of the specimen takes place in a vacuum (0.0001 mm. Hg), the surface structure of the section remains clearly visible even at high temperatures. A magnification of 200-300× can be reached. With this apparatus the following reactions were observed:

1. The α-γ-transformation of pure iron.
2. The transformation of cobalt at 425°C.
3. The solution of pearlite and secondary cementite in pure medium and high-carbon steels.
4. The austenite-martensite decomposition of quenched steel with 2% Mn and 2% C on tempering.
5. The formation of free carbon in white pig-iron on annealing.

The apparatus.

The vacuum mounting for metallographic investigations at high temperatures is shown in section in Fig. 1. The upper part (A) contains the furnace. The space in the oven, which contains the heating coil (G) made of tungsten wire, is turned out of the machinable refractory (E) (Material D 4 of the Staatliche Porzellanmanufaktur Berlin); this is surrounded by a sheet (F). Two springs are cut into the sheet, one to hold the heating apparatus tightly against the upper part, and the other to hold the refractory tightly against the sheet. The heating current passes through insulated terminals (H). The water-cooling leads are shown at (C).

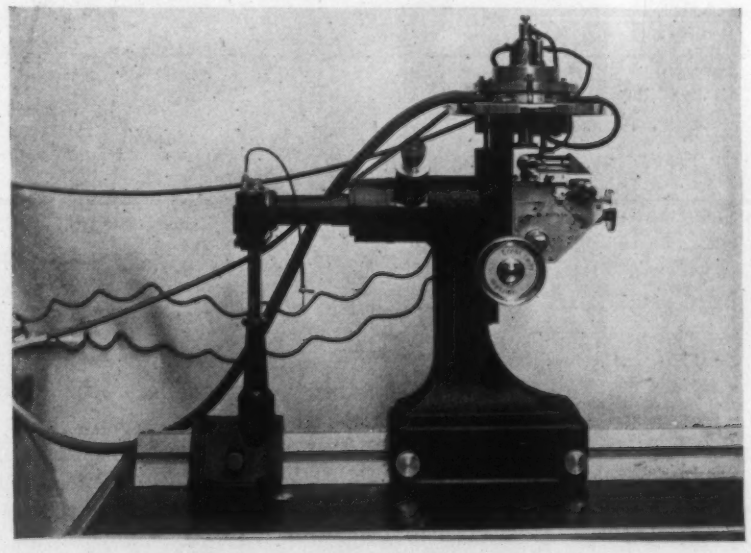

Fig. 2. Apparatus on Microscope with the Leica Camera.

The lower part (B) holds the specimen (P), which rests on the heat-insulating ring (R), made from material D 4, resting on the quartzplate (L). The polished section of the sample can be examined through the quartzplate with the objective (N). The quartzplate is held in the casing with the nut (M) and sealed with a rubber ring. The welded point of a thermocouple (K) lies in a hole in the specimen. A considerable temperature gradient exists in the specimen. In this apparatus, an excellent vacuum is absolutely essential. It was obtained by an oil pump as a first stage and a two step mercury-vapor pump (connected at [D]).

Fig. 2 shows the apparatus in the microscope and the "Leica" camera, which was used in this work. It is possible with the "Leica" to take about 15 pictures per minute, and this camera is a good substitute for a movie. The auxiliary optical apparatus is so arranged, that the focusing for taking a photograph is done by focusing during visual observation.

*Translated and adapted by the authors from the article in *Stahl und Eisen*, Vol. 53, May 18, 1933, pages 532-535.

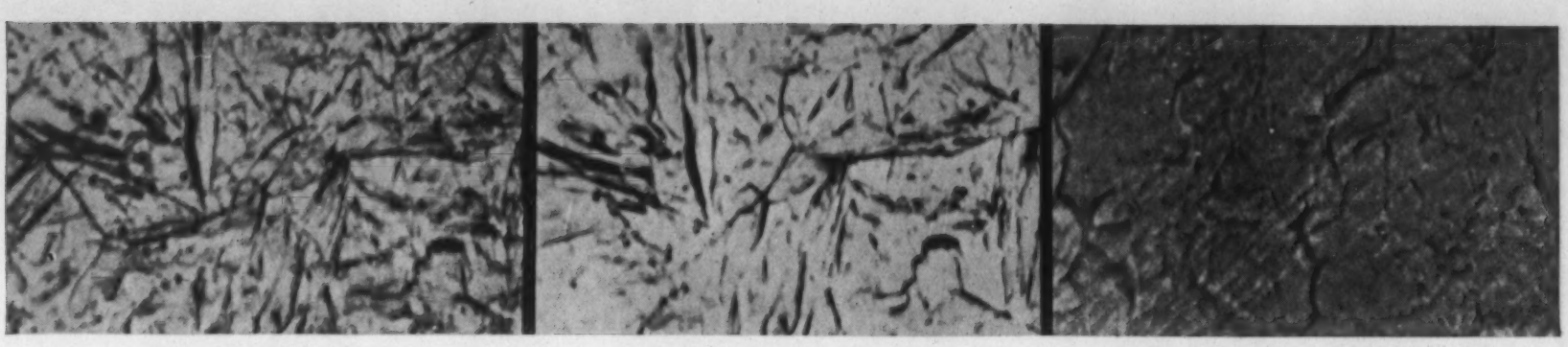

Fig. 3. First Heating above Ac_3.

Fig. 4. After Cooling below Ar_3.

Fig. 5. Second Heating above Ac_3.

Figs. 3-5. Change of an unetched section of pure iron by the alpha/gamma transformation (Magnification 200X).

Results.

α-γ transformation in pure iron:

The metallographic examination at high temperatures was carried out on specimens of carbonyl iron, containing traces of carbon. It was possible to observe on the first heating somewhat below Ac_3 on the unetched surface the development of a network of α-iron crystal boundaries (Fig. 15). If on further heating the $\alpha \rightarrow \gamma$ transformation temperature was passed, then a new network of γ-iron crystals appeared and the boundaries of α-iron became slowly fainter. The appearance of the γ-network is accompanied by a roughening of the surface, which can be ascribed to the change of volume during the transformation (Fig. 3). The observation of Rogers,[2] that the transformation takes place in waves, starting from several points, could not be confirmed. On cooling through the γ-α transformation point a third crystal network appeared, which is connected with the re-formation of α-iron. This third network is sometimes concealed by the formation of a strong surface relief. (Fig. 4.) On heating and cooling once, the section shows a triple network, unless the relief is too strong. Fig. 5, after a second heating above Ac_3, shows the same section as Fig. 4.

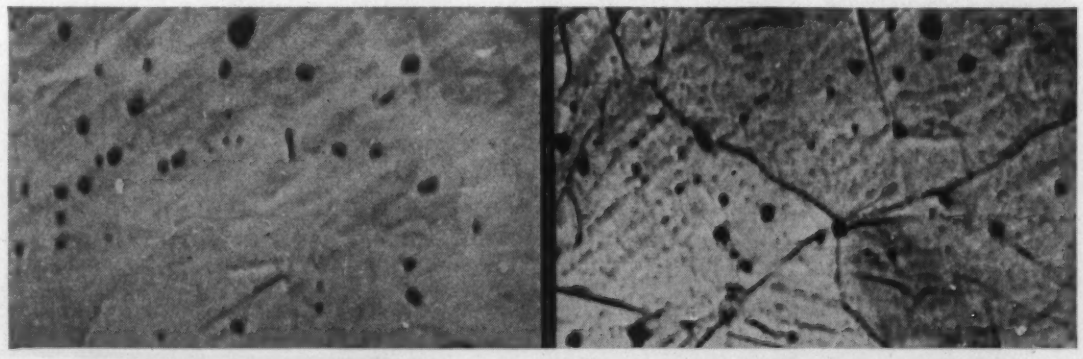

Fig. 6. After heating above the transformation temperature.

Fig. 7. After heating up to 1000°C. and cooling below the transformation temperature.

Figs. 6-7. The Transformation in Cobalt (Unetched Section, Magnification 200X).

Transformation of Cobalt at 425°C.

The transformation of the hexagonal cobalt into the cubic body centered form at 425°C. shows itself by a small relief formation on the unetched surface (Fig. 6). On heating to higher temperatures the grain boundaries slowly appear from about 650° upwards. If the transformation point is passed on cooling, then a needle-shaped relief (slip bands?) is only formed in some crystals (Fig. 7).

Transformation and solubility phenomena in iron-carbon alloys.

The metallographic examination of carbon steels at high temperatures was carried out on lightly etched sections (1% alcoholic HNO_3). The following changes were shown by steels with a carbon content below 0.9%: On reaching Ac_1, the color of the pearlite, which had been obtained on etching at room temperature (Fig. 8), disappeared (Fig. 9). The cause of this probably lies in the solution of the cementite of the pearlite, whose relief, obtained by etching is responsible for the apparent coloring of the pearlite. Slightly above Ac_1 the former islands of the eutectoid were still distinguishable from the ferrite on account of their coarse surface structure. With the completion of the α-γ transformation on further heating, this difference became more and more faint (Fig. 10).

The formation of pearlite on cooling showed itself by a darkening of the corresponding parts of the section, owing to the relief formation during the transformation.

The transformation of steels of about eutectoid composition took place in a small temperature range with the same appear-

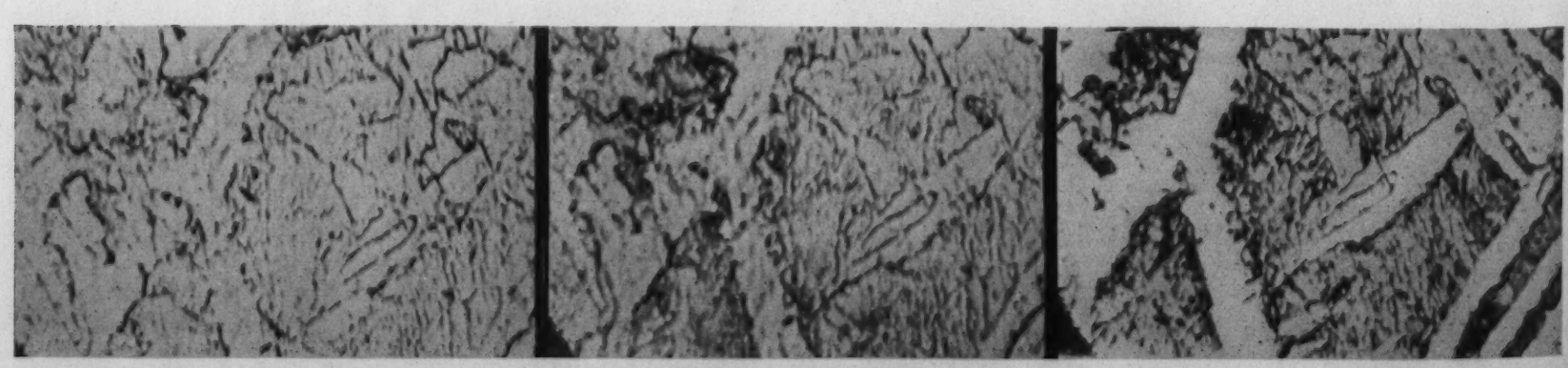

Fig. 8. Etched at room temperature.

Fig. 9. Slightly above Ac_1 Transformation.

Fig. 10. Heated at about 800°C.

Figures 8-10. Change of structure on heating a steel with 0.5% C. (Magnification 200X).

Fig. 11. Etched at room temperature. Fig. 12. Heated up to about 800°C. Fig. 13. Heated to about 1000°C. (Cementite has been dissolved; formation of gamma grain boundaries).

Figures 11-13. Change of structure on heating a steel with 1.5% C (Magnification 200X).

ances, as have been described for the transformation of the pearlite of steels with less than 0.9% C.

With steels above eutectoid composition, it was possible to follow the solution of the cementite in the temperature range between the lines SK and SE of the iron-carbon diagram as well as the transformation of the pearlite and the formation of the γ-grain boundaries above SE (Figs. 11, 12 and 13). The pearlite islands, forming on cooling, often showed straight boundaries, which, as was shown by further etching of the cooled section, were caused by cementite, which partly separated in needle shaped form.

Decomposition of austenite in quenched Mn-steel on tempering.

In a Mn-Steel (2% C; 2%Mn), quenched from 1100°C. in water, the decomposition of austenite was observed on tempering. Hanemann and Wiester[3] have shown, that the transformation of austenite → martensite in carbon steels on quenching takes place with great speed. On the other hand, the decomposition of austenite in the Mn-steel investigated took place very slowly. The formation of martensite on tempering was recognizable by the formation of a needle shaped relief on the carefully polished surface, due to the decomposition of the austenite.

Formation of free carbon in white pig iron.

Finally, we will describe the formation of free carbon on heating white pig-iron, containing 3.8% C and 0.97% Si. This formation of free carbon began above 800°C. first within the ledeburite eutectic and was recognizable by the appearance of small black points in the section. On raising the temperature to about 1100°C., these points increased in size at first slowly and then very quickly, until after reaching a certain size, they did not grow any further. After the conclusion of the carbide decomposition, the free carbon occupied more space on the section, than would be expected from the carbon content of the sample. This phenomenon may perhaps be explained by the fact that the pressure, caused by the formation of free carbon from cementite, is larger in the interior than on the surface of the sample. It is thus possible for the decomposition to take place with greater velocity on the surface. Owing to the rapid decrease in the quantity of carbide in the surface layers, cementite diffuses to the surface, and is here decomposed.

Inclusions of oxygen in steel.

The results of the researches on the amount and the temperature coefficient of the solubility of oxygen in solid iron show great discrepancies. While Oberhoffer, Schiffler and Hessenbruch[4] agree with Benedicks[5,6] on a value of 0.05% O, yet R. Schenck[7] finds a value of 2.05% O at 700°C. and 2.8% O at 1000°C. In a later work,[8] R. Schenck finds a value of 0.4-0.5% O at both 800° and 1000°C. W. Krings and J. Kempkens[9] give a value of 0.11% at 715°C. for the solubility of oxygen in solid iron. Dünnwald and Wagner[10] deduce from the investigation of the equilibria

$$CO_2 + C \text{ (dissolved in } \alpha\text{-iron)} \rightleftarrows 2\ CO$$
and
$$CO_2 + C \text{ (dissolved in } \gamma\text{-iron)} \rightleftarrows 2\ CO,$$

that the solubility of oxygen in solid iron at 800° and 1000°C. is less than 0.01%. J. Reschka[11] examines iron samples, prepared by pressing, sintering and forging of the powdered materials. In these samples oxygen was combined only with iron. Reschka finds, that the solubility of oxygen in solid iron lies below 0.08% O (probably considerably lower), and that the change of solubility up to about 1300°C. is probably very small and should not be more than 0.04% O. It was not possible, to appreciably alter the number and size of the inclusions by annealing and subsequent quenching. H. Schenck and Hengler[12] give with 0.2% O the highest value of recent times, and consider that the A_3- and A_4-transformation temperatures are influenced by oxygen to the extent of about 3°C. H. Esser states in a previous paper[13] that it has not yet been possible to demonstrate convincingly an influence of oxygen on the transformation points of iron. On account of the agreement between his own work and the observation of Reschka, that the inclusions of oxygen were not affected by annealing and subsequent quenching, H. Esser accepted the value of 0.01% (Dünnwald and Wagner) as the most probable.

The fact that the oxygen inclusions in iron do not change their size, form and position in the structure on quenching from high temperatures (900°-1200°C.) strongly indicate, that the solubility of oxygen in solid iron is very small. However there still remains the possibility that this is only apparent, owing to a possible high speed of separation of oxygen. The oxygen inclusions in iron were therefore examined directly at high temperatures with the help of the apparatus, described here.

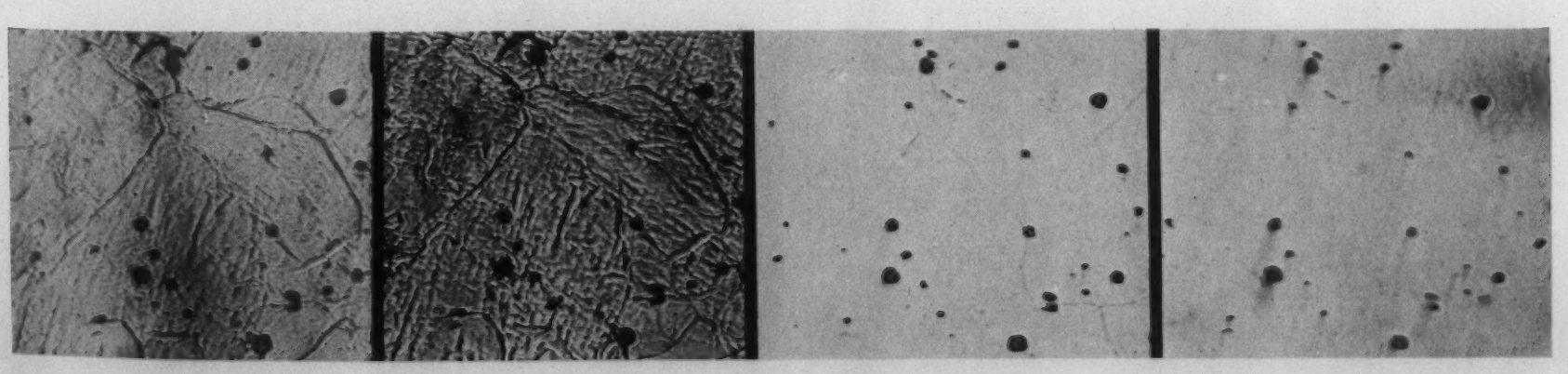

Fig. 14. Room Temperature. Fig. 15. 700°C. Fig. 16. 950°-1000°C. Fig. 17. About 1100°C.

Figures 14-17. Structure of iron with 0.1% oxygen at different temperatures. (Unetched, magnification 200X.)

Two iron-oxygen alloys were used, prepared by the introduction of oxygen into electrolytic iron melted in vacuo.

Alloy 1—Approx. 0.05% oxygen
Alloy 2—0.1% oxygen
0.004% C, 0.004% N_2, 0.02% Mn

Figs. 14, 15, 16 and 17 show the same spot of alloy 2, unetched, 200× at room temperature, 700°, 950°-1000° and 1100°C. The heating to 700° took about 25 minutes. The temperature was then raised to 850°C. during about 10 minutes, held at this temperature for a further 10 minutes, again heated to 950°-1000°C. during 10 minutes and finally raised to 1100°C. during 10 minutes more. The photographs were taken after each of the 10 minute intervals. It appeared that the size of each of the oxygen inclusions did not decrease up to 1100°C., but that they actually increased more than can be explained by thermal expansion. Appreciable solution of the oxide inclusions does not therefore seem to take place. One may perhaps assume that the particles, which are not visible at a magnification of 200× (at 1500× 2-3% more are visible), amalgamate with the larger particles and so cause their growth at high temperatures. (Coagulation of a compound, insoluble in iron, is known in the case of aluminum nitride.) The phenomena which have been described here for alloy 2, were also observed for alloy 1. To determine whether the inclusions really consisted of iron-oxygen compounds, specimens were examined under the microscope during heating in hydrogen. An appreciable reduction of the inclusions took place after a short time at about 700°C. On account of the absence of appreciable quantities of foreign metals, it can be assumed that the inclusions really consisted of iron oxide.

If a pure iron-oxygen alloy is cooled slowly to a temperature slightly below the solidus-line and then cooled comparatively rapidly, the oxygen inclusions are visible in the microscope in an apparently eutectic structure at the grain-boundaries (Fig. 18). This structure can be explained by assuming that the iron crystals, which are formed in the melt on cooling, have during their growth, pushed the fluid FeO-particles to the grain-boundaries. The iron-crystals themselves are practically free from oxygen.

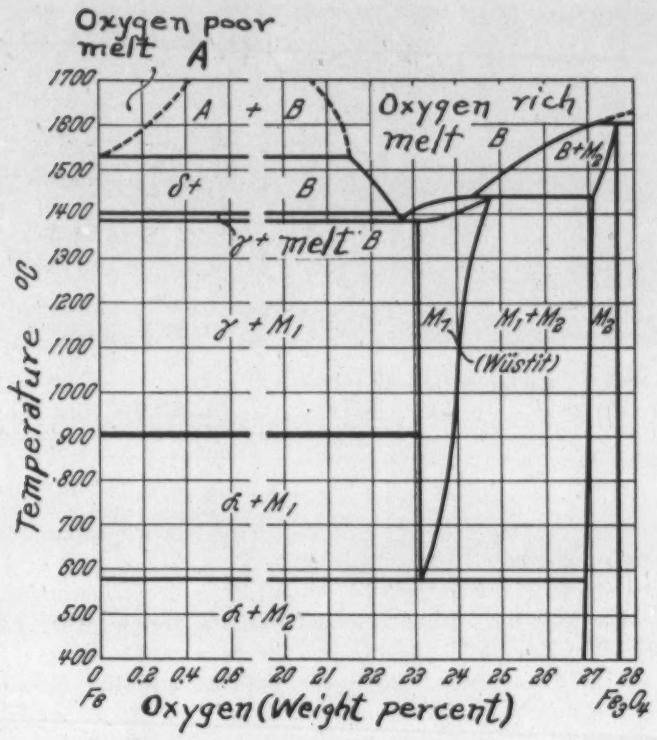

Fig. 19. Iron-oxygen diagram (Between 22 and 28% O) from R. Vogel and E. Martin, Archiv für das Eisenhüttenwesen. Vol. 6, 1932-33, pages 109-111.

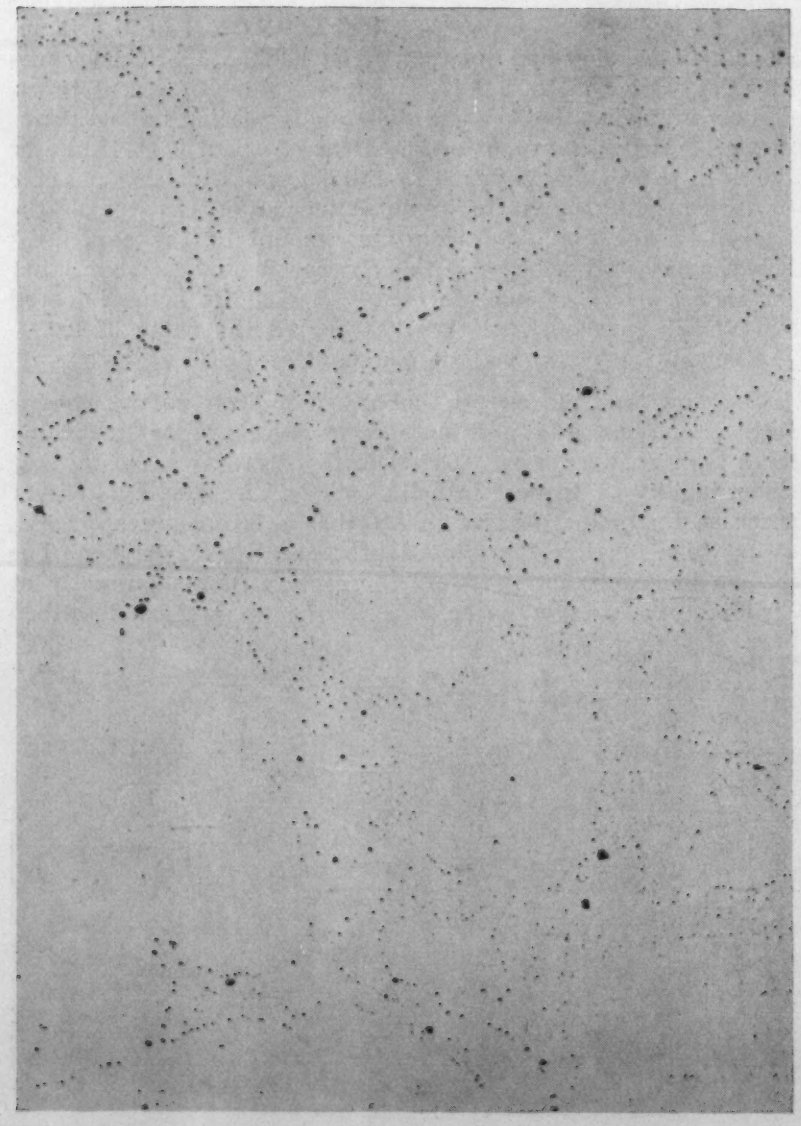

Fig. 18. Apparent eutectic arrangement of oxygen in iron. (Unetched, magnification 250×).

Based on the above microscopic results on the solubility of oxygen in solid iron and the explanation that the eutectic appearance of oxygen in iron is only apparent, the iron oxygen diagram of Benedicks and Löfquist[6] has been altered in the iron rich part so that the area of solid solubility and the eutectic point are obliterated. It is assumed that the area of miscibility in the fluid state extends right up to the pure iron side (Fig. 19).

In the literature there exist several assumptions on the capability of oxygen of forming alloys with iron, which can be hardened by precipitation. From the results of the present work, the necessary conditions for hardening by precipitation are not present in the system iron-oxygen. Insofar as precipitation hardening has been found in oxygen steels, the cause of this must be attributed not to oxygen, but to small quantities of carbon, nitrogen and perhaps hydrogen. It has already been shown for the first two elements, that they can cause precipitation hardening even when present in very small quantities.

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[4] *Stahl und Eisen*, Vol. 47, 1927, pages 1540-1543.

[5] *Zeitschrift Verein deutscher Ingenieure*, Vol. 71, 1927, page 1576.

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[7] *Zeitschrift für anorganische und allgemeine Chemie*, Vol. 166, 1927, pages 113-154.

[8] *Zeitschrift für anorganische und allgemeine Chemie*, Vol. 182, 1929, pages 97-117.

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[10] *Zeitschrift für anorganische und allgemeine Chemie*, Vol. 199, 1931, page 321.

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[12] *Archiv für das Eisenhüttenwesen*, Vol. 5, 1931-32, pages 209-214.

[13] *Zeitschrift für anorganische und allgemeine Chemie*, Vol. 202, 1931, pages 73-76.

HERCULOY

A Copper-Silicon-Tin-Zinc Alloy

by R. A. Wilkins*

An interesting development, in the field of non-ferrous alloys, has been the increasing commercial application of alloys which combine the high resistance to corrosion, characteristic of copper, with physical properties comparable to the steels and similar structural materials.

The alloys, which have commanded interest and which have met with rapidly increasing commercial success and acceptance, have, in general, been characterized by a high copper content with silicon predominant as an alloying constituent. To this basic copper-silicon combination have been added varying percentages of third and fourth constituents for the purposes of attaining desirable characteristics from the view point both of mill fabrication and of ultimate use.

"Herculoy" is the trade name applied to a group of new alloys of the general type outlined above and characterized by the addition of small percentages of tin and zinc, each of which elements causes a distinct and useful modification of the properties of the basic mixture.

The data included in this article have been developed on a specific alloy of the following composition:

Copper	94.75%
Silicon	3.25%
Zinc	1.5 %
Tin	0.5 %

It should be understood that the data presented are based on commercial production, and that while the theoretical mixture is as stated above, minor variations in commercial practice are to be expected.

The presence of zinc in small percentage not only increases the attainable tensile strengths, but has a marked effect on the readiness with which the alloys can be worked either hot or cold. The small amount of zinc appears to have a particularly advantageous action in increasing the facility with which the metal can be cast, and tends to eliminate defects in the wrought products which have their origin in gas or shrink cavities in the original casting.

The presence of zinc may at first glance imply a lowering of the resistance of the basic alloy to corrosive attack. In the alloy under consideration, however, the bearing of the tin on the question of corrosion is of very distinct interest, and the benefits derived from the inclusion of tin more than offset any weakness that might be occasioned by the addition of zinc.

In considering the corrosion resistance of non-ferrous alloys, it is perhaps well to distinguish between outright solution, due to corrosive attack resulting in rapid loss of weight of the metal, and failure, in an engineering sense, because of a combination of mild corrosive attack and internal stresses which results in the physical destruction of the part in question through stress corrosion cracking.

Tin, as an added element in the Cu-Si-Zn series, has a rather pronounced effect on the resistance of the basic alloy to the first type of corrosion mentioned, namely, strictly chemical attack. It has been found that the basic Cu-Si-Zn alloy loses weight in contact with 10% sulphuric acid at the boiling temperature at from 8 to 12 times the rate of the standard mixture containing tin.

A similar resistance to chemical attack, caused by the inclusion of tin, is observed to a certain extent in a comparison of Admiralty metal with ordinary 70-30 brass and Muntz metal with Tobin bronze. The effect of tin is more marked in the case of the alloys under consideration, probably because of the lower zinc content of these latter alloys, which, of course, results in a very much higher ratio of tin to zinc.

In addition, however, to retarding the strictly chemical action of a corrosive agent, the tin, when present, renders it possible to heat treat fabricated forms of the alloy so as to eliminate internal stresses without sacrificing the physical properties attained through cold working.

An alloy of the specific type under consideration will have in the cast or fully annealed condition a tensile strength of approximately 55,000 lbs./in.^{2}, with a high ductility illustrated by elongations under tensile test as high as 70% in 2 inches. Through cold working it is possible, with gradually decreasing elongation and ductility, to increase the tensile strength of such alloys, in commercial size rod, to well over 100,000 lbs./in.^{2} Rod of this type, in which a high strength has been induced by cold working, acquires internal stresses which, if not relieved, lead to the possibility of cracking under mild corrosive attack. The simplest method of relieving such stresses is through a normalizing relief anneal. In the absence of tin it is difficult to carry out such a relief anneal without simultaneously effecting a distinct softening of the material with a consequent lowering of tensile strength.

Herculoy Tanks manufactured by Gerstein & Cooper for Crane Company.

With tin present, however, it has been found that a full relief of all internal stresses can be accomplished at a temperature (550°-600° F.) at which a distinct hardening effect is noted in the alloy.

In commercial practice the introduction of this hardening effect is of great value. It renders it possible to take advantage of the high strengths attainable through extreme cold working, and at the same time to produce a fabricated article which will not be liable to failure through stress corrosion cracking.

In addition to the functions outlined above, the small percentage of tin used has the effect of increasing the tensile strength and the ductility of the basic alloy simultaneously.

The percentage of tin in these alloys, therefore, while small, is of very considerable significance. It ef-

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fects improvements in the basic Cu-Si-Zn alloy which correct the specific weaknesses which have impeded the commercial progress of this otherwise very interesting alloy series.

The susceptibility of being relief annealed, with an increase rather than a decrease in hardness and tensile strength, is such an interesting and distinctive feature of the alloy under consideration that reference to test data will perhaps be of interest.

Specimens of Herculoy wire, drawn from 0.257″ to 0.182″ in 2 passes, were heat treated in a salt bath at 50° intervals from 400° to 700° F. The time of heat treatment was 20 minutes. The resulting variation in Rockwell hardness is illustrated by the curve shown in Fig. 1.

It is interesting to note that specimens ranging from 500° to 600° when bent to a "U" shape and subjected to the standard mercurous nitrate test for 30 minutes showed no tendency to crack even after the displaced mercury remained on the specimen for several days.

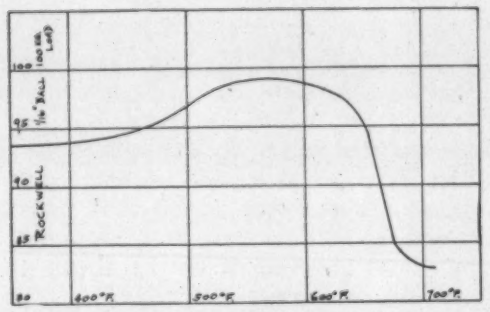


Fig. 1. Herculoy Wire Annealing Test. Drawn from .257″-.218″-.182″. Samples annealed for 20 minutes in salt bath.

The following tests will serve to illustrate the effect of heat treatment on the physical properties of the alloy.

Hot rolled rod was cold drawn from a diameter of 0.750″ to 0.503″. This rod was then cut into 6 specimens, 2 of which were tested as drawn, 2 after a 30 minute relief anneal at 550° F. and 2 after a 5 hour anneal at 550° F.

The test results follow:

		Yield Point lbs./in.²	Ultimate Strength lbs./in.²	Elongation % in 2″	% Reduction in area
As Drawn	1	87,000	135,600	11.0	60
	2	87,000	131,600	12.5	71
30 minutes at	1	104,500	135,000	11.0	55
550° F.	2	105,000	135,000	11.0	55
5 hours at	1	100,000	133,000	11.0	48
550° F.	2	100,000	132,000	10.0	47

The Rockwell hardness B scale, 100 kg. load, 1/16″ ball on the specific specimens tested above rose from 98-99 to 102-103.

It will be observed that the hardening effect of the heat treatment increases the yield point quite materially. An increase in ultimate strength is usually observed after heat treatment, but not to the same percentage extent as the increase in yield point.

In commercial practice a 30 minute relief anneal is used, but the test figures at 5 hours are included to demonstrate the fact that the operation is not delicate and that at the temperature used the time effect is negligible. It is, however, interesting to note that the yield point is being adversely affected by the prolongation of the heat treatment.

The commercial value of the increase in yield point is, of course, obvious.

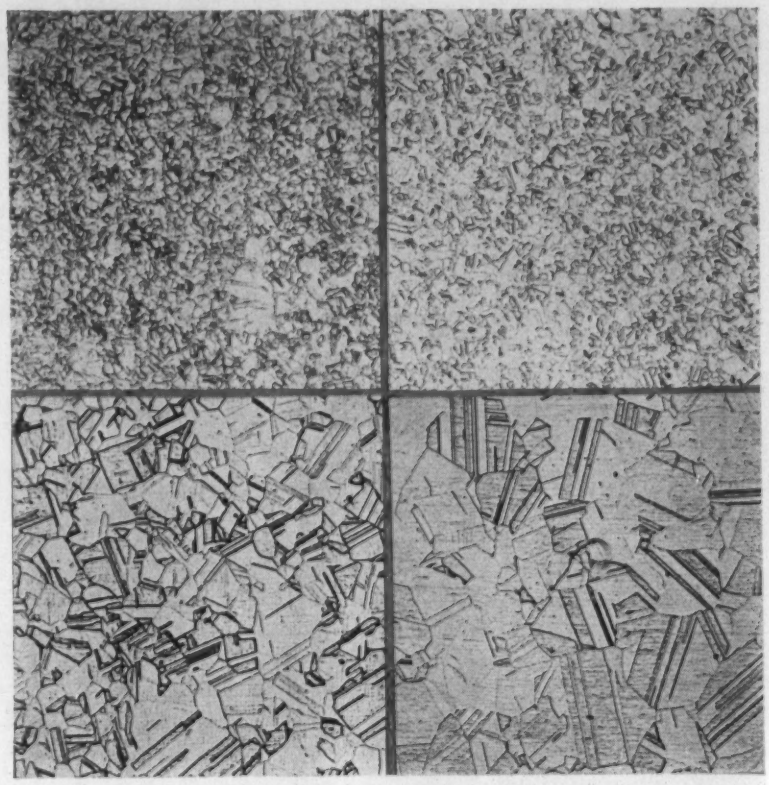
While the inclusion of tin in Cu-Si-Zn alloys in the range indicated is of distinct value in improving the resistance of the alloys to corrosive attack and in rendering possible a heat treatment which establishes an immunity to stress corrosion cracking without loss of strength, it is of additional interest to note that these benefits are not accompanied by a sacrifice of other properties which are desirable in the commercial working or fabricating of non-ferrous metals.

The tin introduces no structural changes and the alloy retains a simple α-structure somewhat suggestive of a 70-30 brass.

The photomicrographs illustrate the typical structure.

Annealed 1 hr. at 1000° F. Air Cooled. Grain Size .010 mm.

Annealed 1 hr. at 1100° F. Air Cooled. Grain Size .015 mm.



Annealed 1 hr. at 1200° F. Air Cooled. Grain Size .065 mm.

Annealed 1 hr. at 1300° F. Air Cooled. Grain Size .085 mm.

Fig. 2. Typical Structures Characteristic of Annealed Herculoy. Etched with $NH_4OH+H_2O_2$. Magnification 75X.

As may be surmised from the chemical composition given and the structure shown, alloys in the range under consideration are susceptible of fabrication and cold working to a high degree. They are readily adaptable to drawing, spinning, or other cold forming operations. They are also readily hot worked, forged, and hot pressed.

Cutting or machining operations are performed with about the same readiness as on mild steel.

To sum up the properties of this type of alloy in a qualitative way, it can be stated that they are structural materials of high strength with a resistance to corrosion which is the approximate equivalent of copper. Their field of usefulness is to a large extent dictated by these basic characteristics.

As would be suspected from the high silicon content of these alloys, their electrical and thermal conductivities are low and the alloys are readily welded by any of the electrical welding methods.

These alloys are very adaptable to sand casting and yield excellent, sound castings which are characterized, of course, by high corrosion resistance combined with remarkable ductility and malleability.

As a quantitative extension of the above general remarks, physical test data on various fabricated forms of the alloy follow:

SAND CASTINGS

Tensile Strength lbs./in.²	Elastic Limit lbs./in.²	Elongation % in 2″	% Reduction in Area
55,000	25,500	40-50	25

ROD

In giving quantitative physical test data on rod, it must be noted that it is beyond the scope of this article to give detailed data on all the possible variations of rod intermediate between the plain hot rolled product and material which is hard drawn to maximum strength.

Figs. 3 and 4 show typical stress-strain curves of the hot rolled alloy and of a representative hard drawn and relief annealed rod.

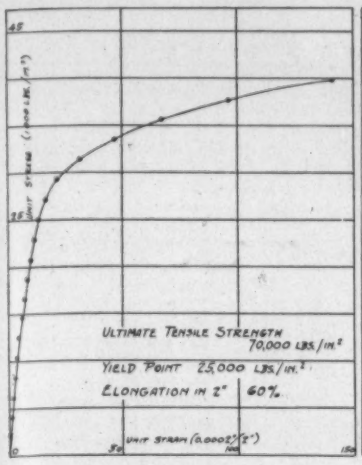


Fig. 3. Stress-Strain Curve for Hot Rolled Herculoy.

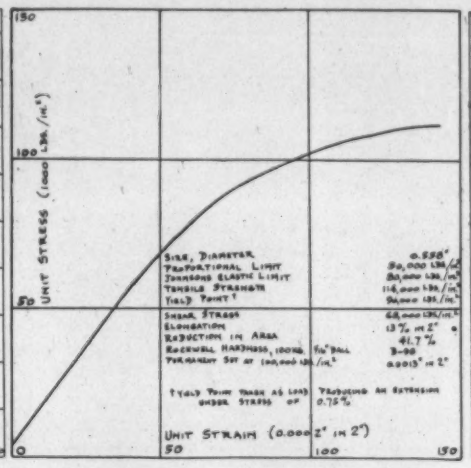


Fig. 4. Herculoy Rod Stress-Strain Curve.

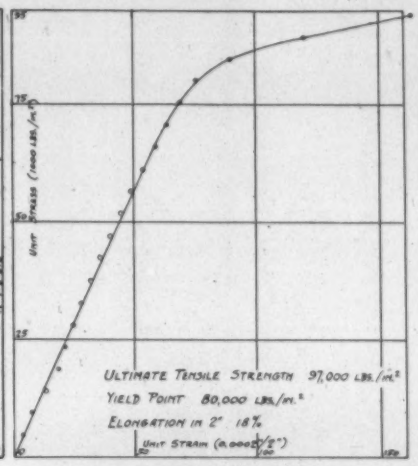


Fig. 5. Stress-Strain Curve. Cold Rolled Herculoy. Four numbers hard 0.100".

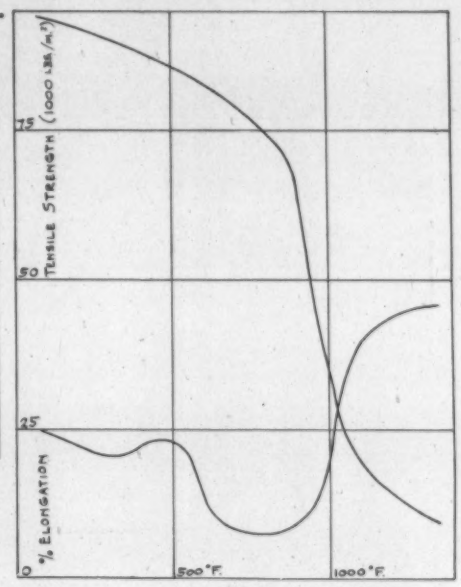


Fig. 6. Herculoy Rod Temperature-Tensile Curve. Samples held at temperature for 1 hour.

Intermediate properties can be obtained through controlling the amount of cold working and in commercial practice properties are so controlled to meet users' requirements.

It should be noted that the modulus of elasticity reaches a maximum with the plain hot rolled material. Where resilience is a determining factor, cold worked material with low modulus and higher strength would be more desirable.

The modulus attainable in hot rolled material is approximately 20 million and this figure gradually diminishes with increasing amounts of cold work until the minimum value of 13 million is reached on full hard drawn stock.

SHEET AND STRIP

Hot Rolled Sheet

In general, the physical characteristics of hot rolled sheet are the same as those for hot rolled bar and the stress strain curve for bar shown in Fig. 3 can be regarded as applicable to hot rolled sheet and plate specimens.

It must be taken into consideration, however, that these tests are conducted on commercial materials and that hot rolled material in the commercial sense has in most instances been subjected to a certain amount of cold work in the final passes. Where welding or hot forming operations on either sheet or rod result in a full anneal of the metal, the residual tensile strength will range between 55,000 and 60,000 lbs./in.² with the former figure constituting a safe basis for engineering computations.

The attainable physical properties on strip material are illustrated by the following table:

Ready to Finish Gage	.045	.050	.057	.064	.072	.080	.090
Finish Gage	.040	.040	.040	.040	.040	.040	.040
Numbers Hard	1	2	3	4	5	6	7
Rockwell	90-91	93	98	97-98	99	99	99
Scleroscope	53	59	63-64	64-65	65-66	67-68	69-70
Brinell	143	158	166	169	177	187	187
Elongation	28.1	18.7/20.3	6.3/9.4	3.1	4.7/6.3	3.1	3.1
Tensile	89,600	95,600	109,100	115,100	128,100	124,200	125,600
	90,500	99,000	111,900	119,400	136,600	125,600	127,400

Rockwell 1/16" B—100 kg.

Fig. 5 shows a typical stress strain curve on cold rolled strip. This curve was based on test specimens 0.100" in thickness and, therefore, will not check with the tabulated data based on 0.040" strip.

Cold rolled and annealed strip and sheet for drawing purposes will show the expected variations in grain size, drawing properties, etc. resulting from the treatment of the metal prior to annealing.

A representative group of properties for cold rolled annealed strip for drawing purposes would be about as follows:

Tensile Strength lbs./in.²	Elongation % in 2"	Rockwell (1/16" B—100 kg.)	Erichsen Cup
66,000	63	48-50	12.5

GENERAL PROPERTIES

Mechanical

The principal mechanical properties of the alloy in fabricated forms have already been given in the form of test figures. The following additional mechanical properties are of interest.

Specific Gravity—8.54.

Impact Value: Energy Absorbed

Standard Izod Specimen—45° V notch.

Hard Drawn Material	25 ft. lbs.
Hard Drawn Relief Annealed	30 ft. lbs.
Annealed specimens bent without fracture	60 ft. lbs.

Fatigue Resistance (standard R. R. Moore specimens—hard drawn relief annealed material):

30,000 lbs./in.² outside fibre stress does not lead to failure under 60 million alternations of load.

20,000 lbs./in.² fibre stress would probably constitute a conservative design figure.

Modulus of Resiliency:

Soft	17.5 in.—lbs./in.³
Hard	375 in.—lbs./in.³

Thermal

Alloys of the type under consideration are not suitable for mechanical use at elevated temperatures. In general, 600° F. is regarded as the maximum temperature at which these alloys should be subjected to mechanical loads.

The curve shown in Fig. 6 illustrates the change in physical properties of tensile test specimens with rising temperature. The specimens tested were held at the temperature of test for one hour before load was applied.

Forging Temperature	1450° F.
Coefficient thermal expansion per °C.—mean 0°—100° C.	0.0000170
Thermal conductivity gram-calories per second per cc. per ° C. between 25° and 125° C.	0.050
Melting Point ° C.	1023.5

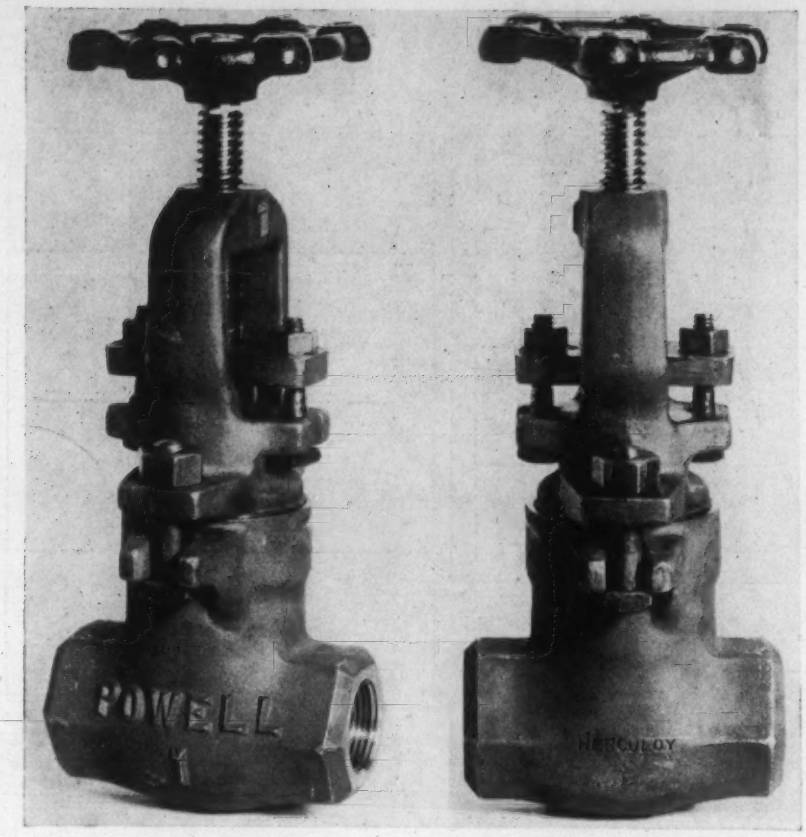
Electrical

Resistivity (soft wire) 21.75 microhms per cc. at 22.5° C.
Max. Induction 470 Maxwells per cm.²
Magnetizing Force 200 Gilberts per cm.²
Normal Residual Induction 100 Maxwells per cm.²
Normal Coercive Force 1.5 Gilberts per cm.²
Temperature Coefficient of resistance per ° C. at 20° C., 0.00046
Permeability (B/H)—2.5

GENERAL APPLICATIONS AND USES

It should be pointed out again that the fields of usefulness of this type of alloy are those in which its properties as a structural material combined with its resistance to corrosion render it an economical material of construction.

It is not the purpose of this article to enlarge on possible fields of application for this material, but a single specific



Herculoy Valves for Foamite Lines, Procter & Gamble Plant, Cincinnati.

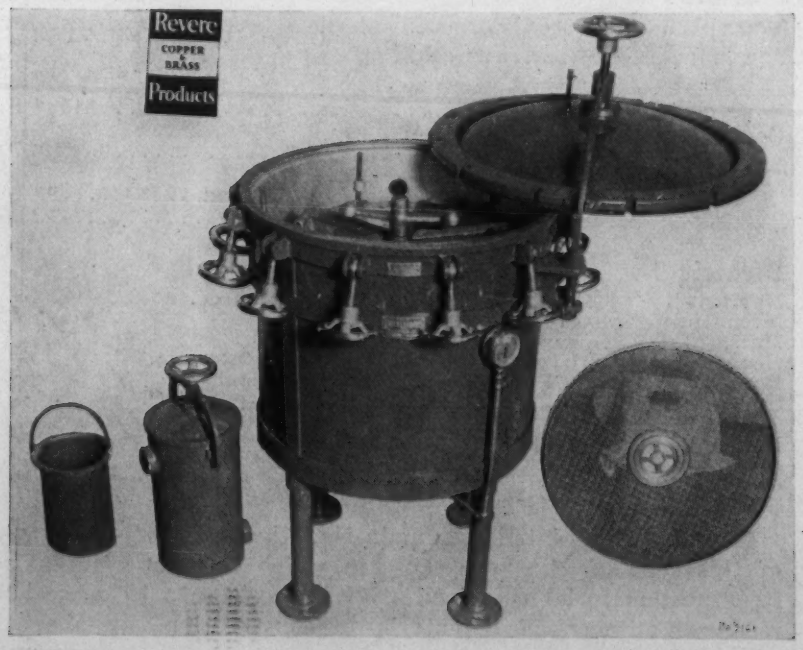
instance of use may be of value in an illustrative way. For the purpose of such illustration, the field of tank construction may be cited. In this field alloys of the silicon bronze type, because of their physical properties, are adequate as materials of construction. In addition, because of the ease with which they can be electrically welded or gas welded, these alloys tend to reduce fabricating costs.

Where the type of corrosive material warrants the use of the silicon bronzes, tanks can be constructed with reference solely to the mechanical and structural requirements. Such tanks can then be used without the further expense in material and labor of protecting the structural material with linings designed to resist corrosion.

The silicon bronzes are resistant to the corrosive attack of a large variety of common commercial reagents and chemicals.

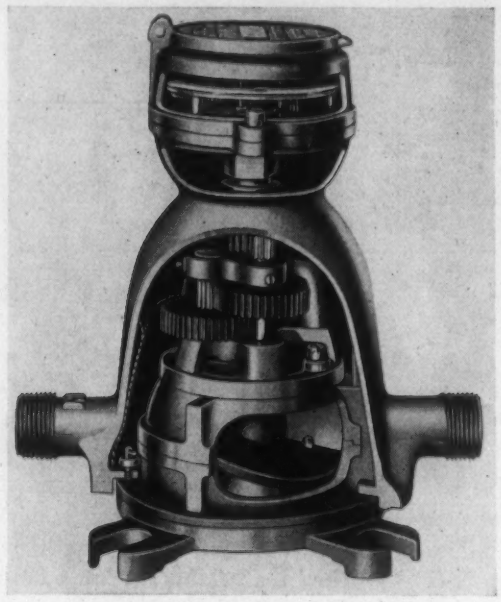
Laboratory and field tests indicate that this type of alloy will give satisfactory service in contact with the following commercial chemicals:

In general, active oxidizing agents cannot be satisfactorily used in contact with the silicon bronzes, and it is, of course,



Tetrachloride Filter made by Bauerle & Morris for American Laundry Machine Company.

Acetic Acid (Very dilute)
Acetic Acid (Moderate dilution)
Acetic Acid (Concentrated)
Acetic Anhydride
Acetone
Aluminum Chloride
Aluminum Sulphate
Ammonia Anhydrous
Amyl Acetate
Amyl Chloride
Benzaldehyde
Bromine
Butyl Acetate
Calcium Chloride
Calcium Hypochlorite
Carbon Tetrachloride
Carbonic Acid
Chlorine Water
Citric Acid
Copper Sulphate
Ethyl Acetate
Fatty Acids
Ferrous Chloride
Ferrous Sulphate
Formaldehyde
Formic Acid
Hydrobromic Acid
Hydrochloric Acid (Very dilute)
Hydrochloric Acid (Moderate dilution)
Hydrochloric Acid (Concentrated)



King Frost Protected Disc Meter for Domestic Service. The disc spindle, gear shafts, pinions and screws are made of Herculoy.

Iodine
Lactic Acid
Magnesium Chloride
Magnesium Sulphate
Oxalic Acid
Phenol
Phosphoric Acid
Sea Water and Brine
Sodium Bisulphate
Sodium Bisulphite
Sodium Carbonate
Sodium Ferricyanide
Sodium Hydroxide (Very dilute)
Sodium Hydroxide (Moderate Dilution)
Sodium Hydrosulphite
Sodium Nitrate
Sodium Phosphates
Sodium Sulphite
Sulphuric Acid (Very dilute)
Sulphuric Acid (Moderate dilution)
Sulphuric Acid (Concentrated)
Sulphurous Acid
Tannic Acid

essential in considering the question of corrosion as affecting any material of construction that the influence of secondary factors such as aeration be considered.

The material herein contained is presented with the thought that the silicon bronzes, of which the specific alloy discussed as a type, will continue to find increasing favor in various industrial fields. It has been the intent of this article to present data and information of general interest in connection with what is substantially a new engineering material.

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Whitman & Barnes, Inc., announces the acquisition of 3 subsidiaries, following consummation of a reorganization plan, ratified by the stockholders of the various companies, on July 21st, as follows: Latrobe Tool Mfg. Co., J. M. Carpenter Tap & Die Co. and Valley Forge Steel & Tool Co.

◈ ◈ ◈

J. C. Fox, Chief Chemist and Metallurgist of the Doehler Die Casting Co., will change his headquarters from the New York Office to the Toledo, Ohio, Plant of the company, where the executive offices are now located.

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The International Convocation on Corrosion sponsored by The Electrochemical Society will be held at the Hotel Stevens, Chicago, Thursday morning, September 7, 1933. A. W. Hothersall of London will discuss the Adhesion of Electrodeposited Coatings to Steel. Karl Daeves of Düsseldorf will emphasize the Effect of Composition and Pre-Treatment of Steels upon the Life of Protective Coatings. E. Herzog of Lille, France, will present his views on the Value of Electrodeposits in Protecting Iron. S. C. Britton and Ulick R. Evans of Cambridge, England, will report on their scientific study of Protective Painting, and Manfred Ragg of Hamburg, Germany, will advocate the Use of Lead Pigments to Protect Iron and Steel. Oliver P. Watts of the University of Wisconsin will present the most modern interpretation of the Theory of Corrosion. K. H. Logan of the Bureau of Standards, Washington, will submit detailed data on Tests Made on Iron Pipes Embedded in Damp Soil. Among others to participate in this International Convocation on Corrosion are Lobry de Bruyn of Amsterdam, Holland, L. Hajda of Dratnova, Czechoslovakia, and F. N. Speller, a member of the Editorial Advisory Board of *Metals & Alloys*, who will act as Chairman of the Meeting.